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Glastonbury, Connecticut

Quality Assurance Project Plan (QAPP)

MacDermid Incorporated
526 Huntingdon Avenue, Waterbury Connecticut

USEPA ID# CTD001164599
Permit # DEP/HWM/CS-151-001

Standard Operating Procedures (SOPs)

Table of Contents

SUMMARY GUIDANCE

General Guidance on Health and Safety

1. Objective

- Personnel performing site investigative activities may encounter known and/or unknown hazards associated with those efforts. GEI has developed a Corporate Health and Safety Program Manual, December 2005 that provides a comprehensive set of procedures to maintain the health of its employees. All employees should be familiar with this document prior to commencing field activities.
- When it is anticipated that potentially hazardous activities are to be conducted, or where there is a potential for contact with hazardous materials or contaminants, a health and safety program must be established, and a site-specific health and safety plan (HASP) must be developed prior to any hazardous site work. Both the health and safety program and the site specific HASP shall comply with 29 CFR1910.120 (b)(1)(iv) and (1)(v) of the OSHA Standard for Hazardous Waste Operations and Emergency Response. A Work Plan may also be required to further define site activities and potential health risks. The project manager should be consulted during the initiation of each new project to determine the necessity of either document.
- All new employees shall complete GEI's Health and Safety Orientation course prior to field assignments.

2. Health and Safety Plan (HASP)

- Site-specific Health and Safety Plan (HASPs) are developed in order to reduce GEI employees' risk of having an accident, becoming ill, or being injured on site. HASPs are used to familiarize site personnel with site conditions; site and project hazards; special procedures; and emergency information. It is the Project Manager's (PM) responsibility to develop a HASP for the majority of projects involving field work.
- A HASP is required for all field work.
- The Project Manager will assign personnel for HASP development. The draft HASP will be submitted to the Corporate Health and Safety Specialist (CHSS) for review and revision of the plan. A due date will be established prior to initiating HASP development.
- Project personnel assigned to develop a draft HASP shall obtain the latest disk version of the appropriate HASP template. Once a draft has been completed, it must be submitted in hard copy form to the Corporate Health and Safety Officer (CHSO) for review and comment. The personnel originally assigned to the draft will perform any revisions to the HASP per recommendation of the CHSO.
- The Project Manager will provide all GEI personnel involved in the project and opportunity to review a copy of the final HASP.

- All GEI project personnel shall review the HASP and sign the HASP signature page to document their reading/understanding of, and willingness to comply with, the requirements of the HASP. The Project Manager shall maintain at the project site and in the project file a copy of the final version of the HASP and the completed signature page.

3. Hazardous Waste Site Work Plans

- To protect the safety of GEI personnel and subcontractors at sites where hazardous chemicals or hazardous waste exist, a site-specific Work Plan describing anticipated cleanup or investigative activities is developed before beginning on-site work. The Work Plan for each site is periodically re-examined and updated as new information about site conditions is obtained.
- The following steps are taken in formulating a comprehensive Work Plan:
  - Review available information, including
    1. Site Records
    2. Waste Inventories
    3. Generator and transporter manifests
    4. Previous sampling and monitoring data
    5. Site photographs.
    6. State and local environmental and health agency records
  - Define work objectives.
  - Determine methods for accomplishing the objectives (e.g. sampling plan, analytical requirements, disposal techniques).
  - Determine staffing requirements.
  - Evaluate the current knowledge/skill level of personnel against the tasks they will perform and situations they may encounter and determine the need for additional training of personnel.
  - Determine equipment requirements. Evaluate the need for special equipment or services, such as drilling equipment or heavy equipment and operations.
  - GEI uses a multidisciplinary approach to the preparation of Work Plans and considers input from all levels of on-site and off-site management, professional, and technical personnel.

4. General Safety Measures

- Levels of protection shall be established for a given site and shall be based upon the best available information regarding known or suspected hazards and the type of planned activity. Activities shall then be performed in accordance with those site-specific levels of protection. Changes in levels of protection should be made only when the level of site specific information improves sufficiently to warrant any change and this change is coordinated through the CHSO and PM.
- The use of respiratory protective equipment shall be in accordance with current OSHA requirements. Air purifying respirator cartridges should be

changed at least once each workday on-site. Only NIOSH/MSHA approved respirators shall be used.

- Eating, drinking, chewing gum or tobacco, smoking or any other practice which increases the tendency for hand-to-mouth contact shall be prohibited within the contaminated zone(s) and prior to washing hands and face within the contamination reduction corridor or decontamination line.
- Medicine and alcohol can intensify the effects of exposure to toxic chemicals. Alcohol, caffeine products and certain medications can contribute to and exacerbate the effects of heat stress. Personnel during site activities should not take prescription and non-prescription drugs when the potential for absorption, inhalation, or ingestion of toxic substances exists, unless specifically approved by a qualified physician. The intake of alcoholic is prohibited during field activities. Caffeine beverages should be avoided during field activities.
- Contact with surfaces known or suspected of being contaminated should be avoided during on-site activities. Avoid walking through puddles, mud, or discolored surfaces; kneeling on ground; leaning, sitting, or placing equipment on drums.
- All personnel connected with a site and engaging in field activities must be familiar with standard operating safety procedures and any additional instructions contained in the Site Safety Plan. Further, all personnel, upon their initial visit to a site, shall read the HASP before performing any site related activities and shall confirm that reading with their signature.
- All field personnel participating in clean-up activities must complete the 40 hour HAZWOPER course and maintained certification through annual 8 hour refresher courses.

5. Reference

GEI Consultants, Inc., Corporate Health and Safety Program Manual, December 2005

6. Contact

- Robin DeHate, GEI Corporate Health and Safety Officer

STANDARD OPERATING PROCEDURE

PM-001 Public Utility Markout

1. Objective

The objective of this SOP is to standardize the utility markout procedures prior to and during excavation. City/state government may have additional requirements for utility markout procedures. Some municipalities/states require that the excavator/consultant perform all markout procedures. All markout procedures should be performed in accordance with local and state regulations.

2. Execution

- The excavator/consultant visits the site, and marks out every place he may be excavating with white paint, flags, or stakes.
- All sample locations should be marked out with sample identification number and type of sample (e.g. boring, testpit, or monitoring well).
- The excavator/consultant fills out all the information about the excavation on a request form specified by state utility markout program and then calls in request. Sample location maps can be faxed to the utility markout program to clarify sampling locations.
- The customer service representative takes the information, and gives the excavator/consultant a mark-out ticket number and a list of utilities notified. The excavator/consultant records these on the mark-out request information sheet for later reference.
- Utilities will only mark out, or clear, facilities under their responsibility. Generally, this means that they will only mark out up to the property boundary. Property owners are then responsible to provide information for private utility locations.
- It is important the excavator/consultant provides an accurate field contact phone number because that is the phone number the facility operators will use should they need to contact you regarding the mark-out request.
- The excavator/consultant then notifies any non-member facility operators if known, such as apartment complexes, commercial complexes, railroads with communication cables, etc.
- Each utility notified either marks out their facilities at the work site, or determines that the site is clear.
- Each utility either completes the mark-out, or notifies the excavator/consultant they are clear, or that they need additional information.
- American Public Works Association (APWA) Uniform Color Code For Marking Underground Utility Lines:

1. **White** – Proposed Excavation
2. **Pink** – Temporary Survey Markings
3. **Red** – Electric Power Lines, Cables, Conduit & Lighting Cables
4. **Yellow** – Gas, Oil, Steam, Petroleum & Gaseous Material
5. **Orange** – Communications, Alarm, Signal Lines, Cables or Conduit
6. **Blue** – Water
7. **Purple** – Radioactive Materials
8. **Green** – Sewers & Drain Lines

- The excavator/consultant then checks off each facility operator on his mark-out request information sheet.
- The excavator/consultant begins work on the scheduled work date and time, (if all the facility operators have responded), taking care to find and preserve any markings that have been made.
- When digging near a buried facility, the excavator/consultant observes the approximate location around that facility.
- If exposing a facility, the excavator/consultant provides proper support and protection for it so that the facility will not be damaged.
- When the excavation is complete, the excavator/consultant provides proper backfill for any facilities that have been exposed, and removes all utility markings.

3. Limitations

- Markout notification time usually do not include holidays. Make sure holidays are considered and markout time is scheduled accordingly. Under no circumstances are markouts allowed to be performed prior to the required markout time.
- Do not use white paint if precipitation is eminent. Consider using stakes if snow is predicted.
- If you need to dig within the approximate location of a combustible, hazardous fluids or gas line (natural gas, propane or gasoline), you must Hand Dig Only! The approximate location is defined as 24" on either side of the designated center line of the facility if the diameter is not provided. Or, 24" from each outside edge if the diameter is provided.
- When excavating close to an underground facility, it is a good practice to have a spotter assist and guide the machine operator.
- Take care not to damage the conduit or protective coating of a facility. If you do, leave the damaged facility exposed and immediately call the utility owner.
- If contact occurs involving gas, the excavator must notify police, fire and emergency personnel, and evacuate employees and general

public. No attempt should be made to tamper with or correct the damaged facility.

- If the excavation work requires significant spans of the facility to be exposed, it is the excavator's responsibility to support them to prevent sagging or collapse as needed. Contact the utility operator for support, guidance, or assistance.

4. References

Connecticut

Name: Call-Before-You-Dig (CBYD)

Telephone: 1-800-922-4455

Website: www.cbyd.com

Wait time after notification: 2 business days (excluding holidays)

Expiration of markout: 30 days

New York State

Name: Dig Safely New York

Telephone: 1800-962-7962

Website: www.digsafelynewyork.com

Wait time after notification: 2 business days

Expiration of markout: 30 days

New York City/Long Island

Name: New York City One Call Center

Telephone: 1-800-272-4480

Website: www.nycli1calldsi.com

Wait time after notification: 2 to 10 days

Expiration of markout: 30 days

New Jersey One Call

Telephone: 1-800-272-1000

Website: www.nj1-call.org

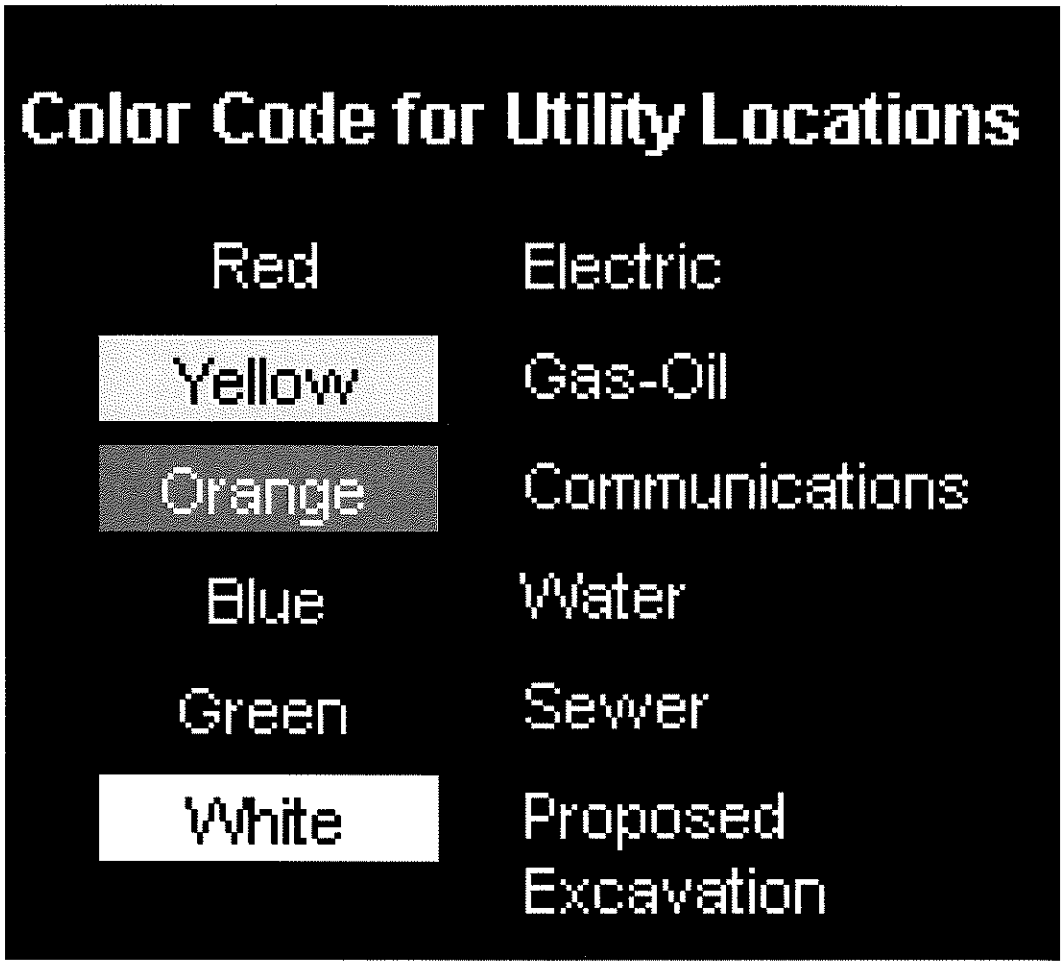
Wait time after notification: 2 business days

5. Attachments

Attachment A – Standard Utility Color Codes

6. Contact

Brian Conte


Color Code for Utility Locations
Red
Electric
Yellow
Gas-Oil
Orange
Communications
Blue
Water
Green
Sewer
White
Proposed
Excavation

STANDARD OPERATING PROCEDURE

RE-001 – Site Reconnaissance

1. Objective

A site reconnaissance is conducted to evaluate the likelihood of contamination at a site that may be attributable to past or present spills, releases, or waste handling/disposal practices. A site reconnaissance should be conducted after available background information is compiled and reviewed. Site reconnaissances are used to confirm, supplement, or modify the existing information about the site.

2. Execution

- Record observations in bound field notebook (See SOP FD-001 Field Notebook or on Site Reconnaissance Form (example provided below).
- Make arrangements with the property owner or occupant for access to the site and site buildings. Be clear that access will need to be provided to all site features.
- Obtain a preliminary base map of the site and a road map with a 500-foot, 1,000-foot, and/or 0.5 mile radius drawn from the boundaries of the site. At the time of the site reconnaissance, determine the street names and numbers at each chosen radii; in rural areas where street numbers are not available, maintain a radius of approximately 1,000 feet.
- If available, review available surficial and bedrock geology, and United States Geological Survey (USGS) maps prior to the site reconnaissance.
- Interview personnel familiar with past and present site conditions during the site reconnaissance. The following information should be recorded: the interviewed person's name, address, telephone number, position in firm or agency, relation to the study site, and years of experience at the site.
- Document the site reconnaissance using photographs. Maintain a photograph log that includes the photograph number, date, location where the photograph was taken, orientation of view, and subject manner.
- Walk entire site property boundaries and make traverses across the site.
- Each of the items on the attached form must be addressed and are described below. If something does not apply to the site, indicate N/A. Do not leave blank.
- Provide general information concerning site identification (street address, size).

- Document site weather conditions, amount of snow, temperature, flooding, etc., in field notebook.
- Topography - Describe general site topography and estimate surface drainage direction.
- Vegetation - Describe general surface vegetation at site. Look for evidence of stressed, dead, or dying vegetation. Changes in the size or age of similar vegetation can indicate areas of clearing, past site disturbance, or former access roads.
- Hydrogeology - Locate surface water bodies and wetlands and, where possible, determine surface flow directions. Identify the following.
- Geology and surface features - Identify landforms, soil exposures, and rock outcrops. Describe the presence and character of artificial fill.
- Monitoring wells - Identify location of monitoring wells on base map and measure distance of monitoring wells from buildings or other permanent structures. If possible, obtain information concerning the type of well and construction details from the client.
- Trace storm drain system , in general, to off-site discharge.
- Land Uses - Describe current and former land uses and history of previous spills or releases. Obtain dates whenever possible. Information may be obtained through interviews with current or former owners, occupants, or employees.
- USTs and ASTs - Describe condition of ASTs and USTs present on site. Look for indications of spills around fill/vent pipes. Look for the presence of fill and vent pipes adjacent to buildings. Inquire about the presence or replacement of former UST and AST locations, size, and contents. Obtain copies of UST monitoring and test records, if available. Identify the number of vent pipes and compare to the number of identified or documented USTs/ASTs. Make note of pavement fracture patterns which may indicate pump islands or UST removal areas. Identify current and former heating sources.
- Waste Information - Describe current operations likely to involve the use, treatment, storage, disposal, or generation of oil and/or hazardous materials (OHMs). Describe the presence and condition of drums, barrels, other storage containers, and disposal areas. Is the area bermed? Are there floor drains present? Record indicators of spills, staining, soil discoloration, leachate breakout, fill materials, or odors. Locate and describe the condition of wastewater systems, pits, lagoons, and disposal areas. If available, obtain copies of Material Safety and Data Sheets for later review. Identify present and past locations where waste is or has been handled or disposed.
- Site Utilities - Describe overhead and underground utilities. Identify whether site is on municipal water or water is supplied through private wells on site. Identify whether site is on municipal sewer or on an on-site septic system, including the location of the disposal area (leach

field, pit, or trench). Inquire whether the site has had a former septic system or wastewater disposal area. Floors should be observed to identify existing or previously abandoned floor drains. Roof drains and grease traps, if any, should be identified. Discharge locations of floor drains, grease traps, roof drains, and catch basins should be identified. Check labels on transformers for polychlorinated biphenyls (PCBs). Note the absence of labels.

- Buildings - Obtain as much information as possible about past and present use activities within the building(s). Describe the condition of floors in the basement or first floor, including cracks and evidence of spills. Describe building construction for example, a slab on grade or basement, steel or wood frame), and note exterior wall construction. Observe building for additions or historical add ons.
- Site Access - Describe fences, roads, topography, vegetation, subsurface or overhead utilities, wet areas, and other factors that may affect site access for a subsurface exploration program.
- Site Abutters - Describe types of general land use activities on abutting properties, including abutters across streets from the site. If possible look for indicators of disturbed land areas and vegetation. Describe general topography and drainage. Look for evidence of USTs. Record names of businesses for regulatory review. Do not trespass on abutting properties.
- Site Vicinity - Identify street addresses at major cross streets up to 0.5-mile radius surrounding the site. Identify sensitive receptors such as schools, nurseries, day care centers, parks, playgrounds, etc., within a 500-foot radius of the site.
- Site Escort - Identify the person who is conducting the site walkover with you.
- Prepare a site sketch or mark the locations of observed conditions on a preliminary base map.

3. Limitations

- If observations are recorded in a field notebook, use the Site Reconnaissance Form (provided below) as a checklist.
- Note any area of the site which could not be observed directly during the site reconnaissance because of restricted access, miscellaneous debris, snow cover, and other adverse conditions.

4. References

ASTM Revised Standards on: Environmental Site Assessments, (2005), E 1527-05

Code of Federal Regulations All-Appropriate Inquiries Standards and Practices for All Appropriate Inquiries" (40 CFR Part 312)

Guidance to Environmental Site Assessment (September 1992), National Ground Water Association.

Standard References for Monitoring Wells (January 1991), The Massachusetts Department of Environmental Protection, DEP Publication #WSC-310-91.

The Massachusetts Contingency Plan (July 30, 1993), The Massachusetts Department of Environmental Protection, 310 CMR 40.0483.

5. Attachments

Attachment A - Site Reconnaissance Checklist

6. Contact

Mr. Gary Iadarola

RE-001 Attachment A: Site Reconnaissance Checklist

GEI Job No.:

Date:

Site Reconnaissance performed by:

GEI Project Manager:

Part 1: General Information

Client

Property Name

Address

Size of Site Easements

Paved parking lot areas (patched areas)

USGS Quadrangle GIS Maps

Available bedrock or surficial geology maps

Weather at time of visit (include snow cover, flooding, etc.)

Part II: Topography (level-rolling-hilly-etc.)

Describe

Degree of slopes, approx.

Drainage (estimated surface water flow direction)

Part III: Surface Vegetation (wooded-brush-grass-landscaped)

Describe

Stressed or stained vegetation

Describe

Part IV: Hydrogeology (surface water bodies and wetlands)

Describe

a. Geology (bedrock outcrops, fill areas, soil exposures, trenches, etc.)

Describe

b. Surface features (drumlins-valley floor-flood plain)

Describe

c. Monitoring wells (number, size, roadbox, stand-up pipe, location, distance from buildings)

Describe

Part V: Land Use (current, former)

Describe

Part VI: USTs and/or ASTs

Vent and fill pipes (number and locations)

Tank ID	UST/ AST	Capacity (Gallons)	Date Installed	Date Removed	Date of Last Tightness Test

Part VII: Waste Information (drums, barrels, storage containers, wastewater systems/discharges, pits, lagoons, odors)

Describe

Dumpsters/Trash Collection (past & present)

Waste Materials

Dumped Material or Miscellaneous Debris

Areas of Fill

Part VIII: Utilities

Water Electric Gas Oil Cable/Phone
Sewer Septic Leachfield Catch Basins
Manholes Drywells Transformers

Describe

Part IX: Buildings (exterior)

Number of Buildings/Additions

Locations

Size of Buildings

Number of Stories

Age

Describe

Part X: Buildings (interior) (Fill out this section for each building interior.)

Inside Building Descriptions and Uses (offices, commercial, industrial, manufacturing, and other)

Chemical Storage

Basement and/or 1st Floor (type of construction)

Floor Drains, Sumps (number and location)

Floor Condition (cracks, spills)

Part XI: Site Access (fencing, gates, roads, etc.)

Describe

Part XII: Site Abutters (addresses and land uses)

North

East

South

West

Part XIII: Site Vicinity

Names of Cross Streets

Direction in which Street Addresses Increase or Decrease (within 1,000-foot radius of site)

Schools, nurseries/day care centers (within 500-foot radius of site)

Part XIV: Site Escort

Affiliation

Employed at facility

Part XIV: Additional Comments:

STANDARD OPERATING PROCEDURE

FD-001 Field Notebook

1. Objective

Proper documentation of all site activities is a crucial part of the field investigation process. Documentation, relative to sampling procedures, includes sample labels, sample seals, field logbooks, chain of custody records, sample analysis request forms, and laboratory sample logs. The field notebook serves as a record of significant field activities performed or observed during the project. The field notebook provides a factual basis for preparing field observation reports, if required, and reports to clients and regulatory agencies. Example field notes are provided in Appendix A.

2. Execution

- Use a separate all-weather bound notebook for each site/location/project number.
- Write neatly using black or blue waterproof pen (or note if field conditions [i.e., cold or wet weather] require use of pencil).
- Write the project name, project number, book number (i.e., 1 of 3), and date on the front cover. On the inside cover, identify the project name, project number, and "Return Book To:" the office address of the project manager.
- Number all of the pages of the field book starting with the first entry.
- Record activities as they occur.
- Neatly cross out mistakes using a single line and initial them. Erasures are not permitted. If an error is made on an accountable document assigned to one individual, that individual will make all corrections. The person who made the entry will correct any subsequent error discovered on an accountable document. All subsequent corrections will be initialed and dated.
- Sign or initial and date the bottom of every page with an entry. outplace a diagonal line through unused portions of a page.
- Record the following information upon each arrival at the site:
    1. Date/time/weather/project number
    2. GEI personnel
    3. Purpose of visit/daily objectives
    4. Record conversations with: [Recommendation - If possible, record telephone numbers of individual contacts for the site in the field notebook.]
    5. Contractors
    6. Clients
    7. Visitors (include complete names, titles, and affiliations whenever possible).

8. GEI office staff
9. Landowners (site or abutters)
10. Note time of arrival and departure of individuals visiting the site.

- Type and quantity of monitoring well construction materials used
- Use of field data sheets or electronic logging equipment (e.g. boring logs, monitoring well sampling logs, etc.)
- Ambient air monitoring data
- Locations and descriptions of sampling points
- Sample media (soil, sediment, groundwater, etc.)
- Sample collection method
- Number and volume of sample(s) collected and sample bottle preservatives used
- Sample identification number (s) and date and time of sample collection
- Approximate volume of groundwater removed before sampling
- Field observations
- Any field observations made such as pH, temperature, turbidity, conductivity, water level, etc.
- References for all maps and photographs of the sampling site(s)
- Information pertaining to sample documentation: bottle lot numbers/ dates, method of sample shipments, chain-of custody record numbers, and overnight shipping numbers.
- Surveying data (including sketches with north arrows)
- Changes in weather
- Rationale for critical field decisions
- Recommendations made to the client representative and GEI Project Manager
- Include a site sketch or representative site photograph of conditions at the end of the day, if required.
- Time
- Summarize work completed/work remaining
- Place a diagonal line though and sign portions of pages not used or skipped.
- Bottom of each page signed and dated.

3. Limitations

- Only record facts.
- Allow time at the end of the day to write your journal, and make it a priority, even at the expense of observing time.
- Record all observations regardless of relevancy.
- Identify conditions or events that could affect/impede your ability to observe conditions.

- Do not use spiral notebooks because pages can be easily removed.

4. References

New Jersey DEP Field Sampling Procedures Manual, August 2005

Yerington Mine Site SOP-03 Standard Operating Procedure Field Notes and Documentation, Revision 0 Revision Date: June 6, 2006

ASFE Model Daily Field Report (1991), ASFE, Inc.

5. Attachments

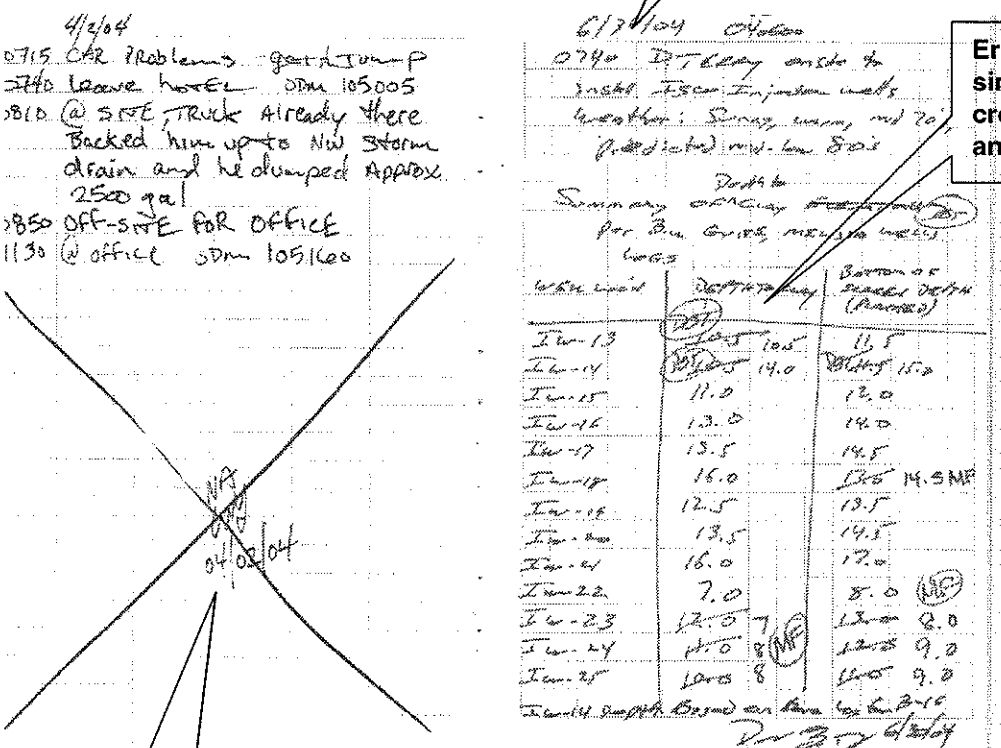
Attachment A - Example Field Notes

6. Contact

Melissa Felter

SOP FD-001

Attachment A – Example Field Notes



4/2/04
0715 CAR Problems - get a Jump
0740 Leave hotel SDM 105005
0810 @ SITE, TRUCK Already there
Backed him up to NW Storm
drain and he dumped Approx
2500 gal
0850 OFF-SITE FOR OFFICE
1130 @ office SDM 105160

6/7/04
0740 Drive to
Install Test Injection wells
Weather: Sunny, warm, mid 70's
(predicted) mid-to-low 80's

Summary of Drilling Progress for 3 in Grids

Well Grid	Depth to Clay	Bottom of Sand Depth (Planned)
Iw-13	10.5	11.5
Iw-14	14.0	15.0
Iw-15	11.0	12.0
Iw-16	13.0	14.0
Iw-17	13.5	14.5
Iw-18	16.0	14.5 NF
Iw-19	12.5	13.5
Iw-20	13.5	14.5
Iw-21	16.0	17.0
Iw-22	7.0	8.0 NF
Iw-23	12.0	13.0 8.0
Iw-24	11.0	12.0 9.0
Iw-25	10.0	11.0 9.0

Iw-14 Depth Based on Iw-16 & Iw-18

NA
04/02/04

Start of each day includes:
Date
Project Number
People on site
Purpose of Work
Weather Conditions

Errors are single line crossed out and initialed

Blank Space Crossed out and initialed

Bottom of each page signed and dated

GEI Consultants

STANDARD OPERATING PROCEDURE

FD-003 Sample Handling and Chain of Custody

1. Objective

To properly collect, label, document, preserve, package, transport environmental samples, and to provide a record of the custody of any environmental field sample from time of collection to delivery to the laboratory. The Chain-of-Custody (COC) can be used as a legal document to guarantee that samples were not mishandled and that they were delivered to the laboratory within the timeframe necessary to start analysis. A sample is under custody if:

a) it is in GEI's possession; or
b) it is in GEI's view after being in GEI's possession; or
c) it was in GEI's possession and then it was locked up to prevent tampering; or
d) it is in a designated secure area. GEI facilities are designated secure areas.

2. Execution

- Review the work plan prior to sampling to determine the following:
  i. The analysis required by the period and sample volumes required by the laboratory to perform those analysis. (Be explicit when requesting analysis on the COC (e.g. rather than "VOCs" write "VOCs 8260")
  ii. The turnaround time required by the project
  iii. If the data will be sent directly from the laboratory to the data validator or Data Group
  iv. Holding time restrictions for sampling media and analytical methods
- Label the jar or bottle not on the cap.
- Following sample collection, the sample container is labeled using a waterproof marker with the sample ID, the date and time (military time) of sample collection, project number, sample preservatives, and the sampler's initials. Sample custody begins at this time.
- Record the above information in the Field Notebook.
- Individually wrap sample jars with packing material. Place samples in a chilled (4°C) cooler immediately after collection.
- Complete a chain of custody (COC) for the samples as described below, and sign off on the COC each time a new person takes possession of the samples. A COC form must accompany each shipment/delivery of samples to the laboratory. GEI or Laboratory COC forms may be used as long as the laboratory form contains the same required information as described below.

- Place a custody seal on the cooler if shipping. Transport samples to the laboratory as soon as possible.

2.1. Chain-of-Custody (COC) Completion

- Record the project name and number, the sampler's name(s) and the state where the samples were collected.
- For each sample, enter the sample identification number, date and time (military time) collected, whether the sample is a grab or composite sample and the number of sample containers. Record the type of analysis (including laboratory method; e.g. EPA-SW846 Method XX) requested and the preservative (if appropriate) in the vertical boxes.
- When samples are ready to be relinquished, complete the bottom of the form with date and time (military time) and signatures of relinquisher and receiver of samples as indicated. The sample collector is always the first signature while the analytical laboratory is the final signature. Theoretically, all individuals handling the samples between collection and laboratory should sign the form; however, if a common carrier (i.e., Federal Express, UPS) is used for shipping, GEI must identify the carrier in the 'Received by' box on the COC. If the sampler hand delivers the samples to the laboratory, the received box must be signed by the laboratory.
- The forms are in triplicate (white, yellow, and pink copies). The pink copy should be retained by the sampling personnel and provided to the Data Group for proper filing. The white and yellow copies should accompany the samples to the laboratory.
- Prior to sample shipment, the COC must be placed inside the cooler (in a ziplock bag or other watertight package taped inside the lid of the cooler), and the cooler must be sealed with a signed COC seal.
- If a common carrier such as FedEx is used to transport the samples to the laboratory, include the carrier tracking number and identify the carrier in the "Received by" box on the COC.
- Any unused sampling containers/media that is sent back to the lab should be included on the COC. Return samples to the laboratory in a timely manner.
- Field duplicates should be anonymous to the laboratory, but must be recorded for use by the Data Group. To keep track of this information, link the field duplicate with the proper sample in the field copy of the COC and also the field book.
- After the samples are sent to the laboratory, the field copy must be sent to the Data Group. You can send the field copy with duplicate information in the mail to Data Group.

3. Limitations

- The field notebook must document all GEI personnel who had custody of any samples prior to shipping the samples to the laboratory, the samples must be relinquished to the shipper and the COC signed and dated by the sampler and the shipper, even if both people are GEI personnel.
- Keep the number of people involved in collecting and handling samples and data to a minimum.
- Only allow people associated with the project to handle samples and data.
- Always document the transfer of samples and data from one person to another on chain-of-custody forms.
- Always accompany samples and data with their chain-of-custody forms.
- Give samples and data positive identification at all times that is legible and written with permanent ink.
- When sending samples via a common carrier, use one COC per package.
- Do not send samples from more than one site with separate COCs in a single package.

4. References

New Jersey Department of Environmental Protection, Field Sampling Procedures Manual, August 2005.

Connecticut Department of Environmental Protection, Guidance for Collecting and Preserving Soil and Sediment Samples for Laboratory

Determination of Volatile Organic Compounds, Version 2.0 February 28, 2006.

5. Attachments

None

6. Contact

Mrs. Melissa Felter

STANDARD OPERATING PROCEDURE

FD-004 Photodocumentation

1. Objective

To properly document and retain photographic records. Keeping a record of photographs taken is crucial to their validity as a representation of an existing situation.

2. Execution

- Photographs of a site, individual samples, or other observations should be taken using a digital camera.
- All photographic records should be recorded in the Field Notebook (SOP FD 001) and the following information should be recorded in the field notebook.
- Number of photograph in sequence
- Compass direction describing the direction the photograph was taken (e.g. looking southeast)
- Brief description of what the photograph is intended to show
- The field notebook should also note who took the photographs, and the date and time each photograph was taken.
- The photographs should be electronically backed up on a computer or other data storage device.
- Photographs should be placed on a photograph record template and the relevant information describing the photograph should be inserted into the caption section for each photograph.

3. References

New Jersey Department of Environmental Protection, Field Sampling Procedures Manual, August 2005.

4. Attachments

Attachment A - Photodocumentation Template

5. Contact

Melissa Felter

GEI Consultants, Inc.

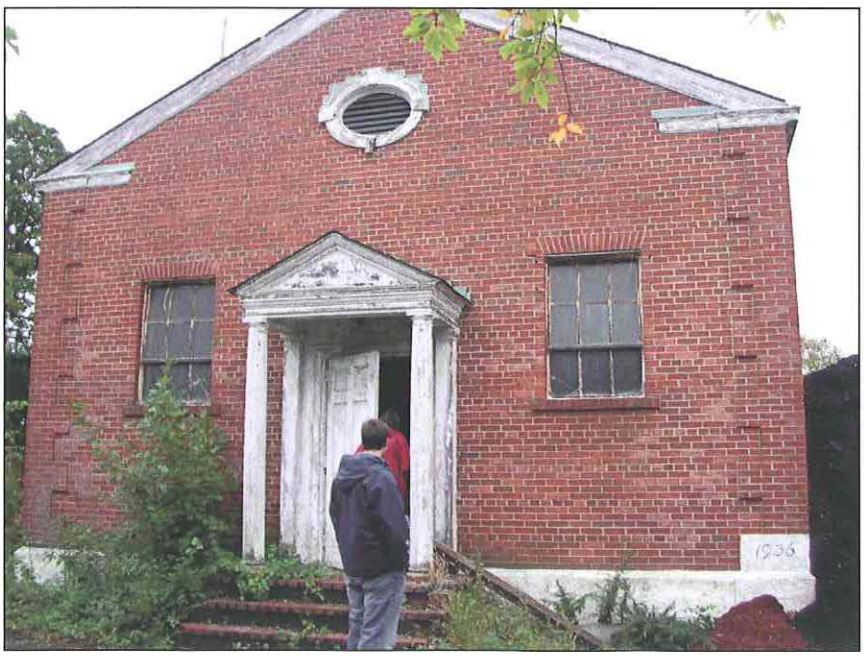
PHOTOGRAPHIC RECORD

Project: Project Name

Location: Project Location

Photographer: K. Barber
Date: 10/25/07
Photo No.: 1
Direction: N

Comments:
Entrance of site with tree mulching operations.



Photographer: K.Barber
Date: 10/25/07
Photo No.: 2
Direction: W

Comments:
On-site building built in 1936.

STANDARD OPERATING PROCEDURE

DM-002 Hollow Stem Auger

1. Objective

To standardize the drilling of overburden soil using hollow-stem augers.

2. Execution

- Contact the Owner to determine the locations of underground utilities/obstructions. Verify with the contractor that the utility clearance service of the particular state you're working in has been contacted. Ask the subcontractor to provide you with the utility clearance authorization number and the time of clearance to proceed, and record the number in the field notebook (See SOP PM-001).
- Inspect the drilling rig to make sure it is clean and that the down-hole equipment has been steam-cleaned. Check that the steam cleaner is working properly (i.e., that steam is being produced). Measure and record lengths of all down-hole drilling equipment, including the drilling heads and miscellaneous rods and attachments. Record all observations and measurements in the field notebook.
- If a surface-soil sample is desired, collect this sample with a split-spoon sampler prior to setting the first flight of augers up over the borehole. For all soil samples, use a 140-lb hammer to drive the sampler, unless conditions necessitate using a 300-lb hammer. Count and record the number of blow counts per 6-inch increments (confirm blow counts with driller if necessary). Split-spoon sampling should be conducted in accordance with SOP SM-001 Soil Sampling.
- Decontaminate the split-spoon sampler after each use (see *Equipment Decontamination,* SOP QA-001) or use another decontaminated split-spoon sampler.
- Direct the drillers to drill the borehole to the top of the next sampling interval. Remove the auger cutting bit/plug and insert the split-spoon sampler into the interior of the augers (the drillers are responsible for this activity). Measure the stick-up of the rods attached to the sampler to ensure that the nose of the spoon is in virgin soil below the augers.
- Watch for signs of a soil strata change at depth during drilling (i.e., change in blow counts, change in soil color, soil wetness, soil contamination, bouncing of the drill rig, etc.). If important to the investigation, stop drilling and collect a soil sample.
- Repeat until the borehole has been drilled to the desired depth.
- If a monitoring well is not installed in the soil boring, fill the boring with either cement/bentonite grout or properly-tamped and hydrated bentonite. Do not backfill the boring with drill cuttings unless explicitly allowed under state-specific regulations.

- Complete boring log and, if necessary, well installation logs (SOP SS-004, *Soil Classification*).
- Record boring locations on a site map and in a field notebook sketch. Measure each location from on-site reference points in the field notebook so that enough information can be obtained to recreate the location.

3. Limitations

- At all times, follow safety procedures as defined in the site-specific Health and Safety Plan.
- In areas of significant soil contamination, hollow-stem augers may cross-contaminate upper soil layers as contaminated cuttings move up the auger flights. The potential also exists for contaminated augers to carry contamination to deeper soil strata.
- If *in situ* borehole permeability tests are to be performed prior to installation of the monitoring well, the hollow-stem auger method is not appropriate due to water loss at the auger junctions.
- If significant unanticipated contamination is encountered during drilling, stop drilling to confer with the project manager and evaluate health and safety conditions. If the borehole is to be advanced below the contaminated strata, use telescoping techniques (see SOP DM-007 *Borehole Telescoping Techniques*) to avoid cross-contaminating underlying geologic strata.
- When drilling below the groundwater table in fine to medium sands, the potential exists for the phenomenon of "running sands" or "blow in" to occur. Frequent measurements inside the hollow-stem augers after the drill bit/plug is removed will indicate if running sands are present. If sands start to flow into the auger, pour clean water into the augers and keep the augers filled during sampling.
- If necessary, arrange for the storage of contaminated soil cuttings and water in drums or other appropriate containers in a secure place at the site. Containers should be labeled.
- Plan the drilling program to drill borings from the least- to most-contaminated areas. Be prepared in advance and know where alternative drilling locations are in the event that problems are encountered at each planned soil boring location. Alternative locations will need to have utility clearance.

4. References

Standard Practice for Design and Installation of Ground Water Monitoring Wells in Aquifers (October 1990), American Society for Testing and Materials [ASTM] D5092-90.

Nielsen, D.M. (1993), "Correct Well Design Improves Monitoring," Environmental Protection, July, pp. 38-49.

Standard References for Monitoring Wells (April 1991), Commonwealth of Massachusetts Department of Environmental Protection, WSC-310-91.

5. Attachments

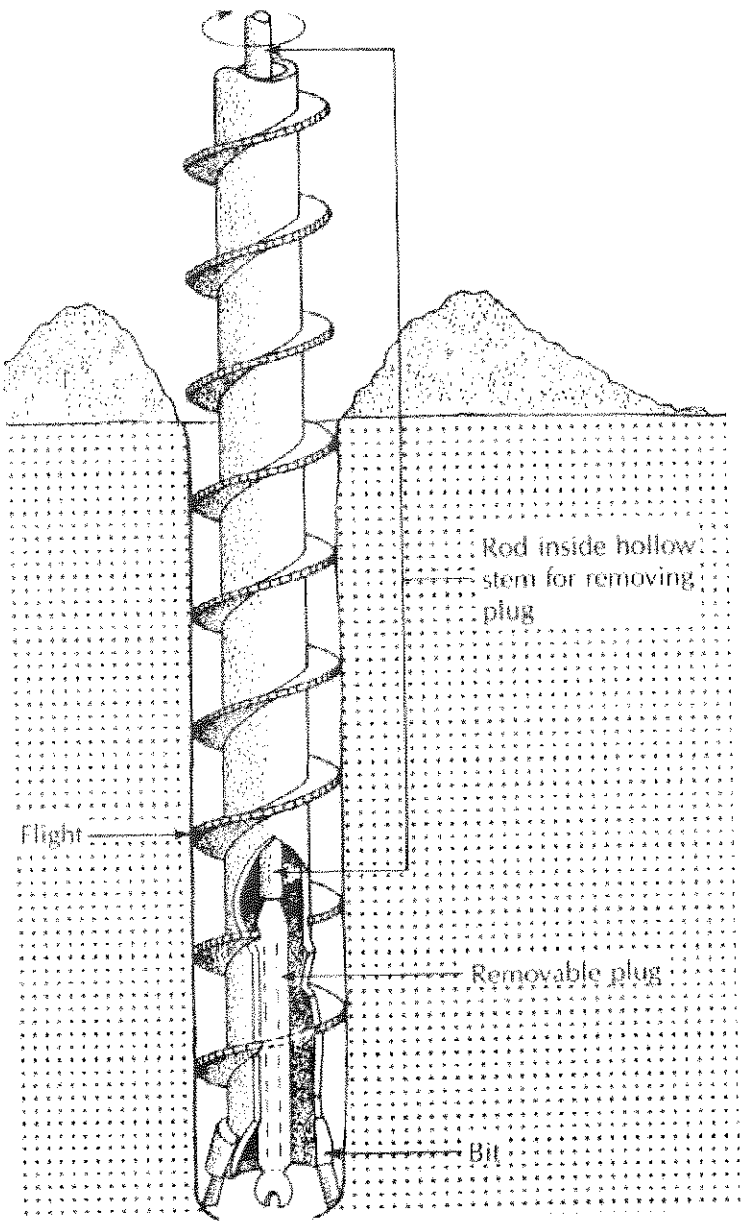
Attachment A – Hollow Stem Auger

6. Contact

Mr. Lynn Willey

SOP DM-002

Attachment A – Hollow Stem Auger



GEI Consultants

STANDARD OPERATING PROCEDURE

DM-004 Sonic Drilling

1. Objective

The objective of this SOP is to standardize the drilling of overburden soil borings for environmental investigations. This SOP addresses the use of sonic drilling to drill the soil boring.

2. Execution

- Contact the Owner to determine the locations of underground utilities/obstructions. Verify with the contractor that the utility clearance service of the particular state you're working in has been contacted. Ask the subcontractor to provide you with the utility clearance authorization number and the time of clearance to proceed, and record the number in the field notebook.
- Inspect the drilling rig to make sure it is clean and that the down-hole equipment has been steam-cleaned. Check that the steam-cleaner is working properly (i.e., that steam is being produced). Measure and record lengths of all down-hole drilling equipment, including the drilling heads and miscellaneous rods and attachments. Record all observations and measurements in the field notebook.
- Collect soil cores in shorter runs. While some sonic rigs have the capability of collecting 20 feet of soil core at a time, the process of collecting the longer core results in the core being in contact with the core barrel for a longer period of time and consequently absorbing more heat from the core barrel itself.
- The core barrel should be cleaned with tap water following each use.
- The field geologist will classify and sample the soil located within the liner.
- Upon completion, the excess soil will be placed into a 55-gallon drum for disposal and the inner liner properly disposed.
- The core barrel will then be advanced within the isolation casing on the same borehole to collect the next soil core interval.
- Add water between the inner core barrel and the outer override casing. This water would reduce friction and adsorb heat between the inner core barrel and the outer over ride casing.
- Maximize drilling advance rate. The faster the core barrel is advanced, the less likely the core barrel will heat up, and the less contact time the soil core has with the core barrel. Drilling with a 3-inch diameter core barrel and a 5-inch diameter override casing, instead of the standard 4-inch core barrel and 6-inch over ride casing, may increase advance rates and reduce the potential for soil core heating.

- If a significant decrease in drilling advance rate is observed, stop drilling and remove what soil core has accumulated in the core barrel. Resume drilling through the resistant material (gravel, boulder, hard clay, etc.). When the resistant material has been penetrated and the drilling advance rate increases, stop drilling and remove what material has accumulated in the core barrel.
- Wash down the core barrel with cool water to cool the core barrel and associated casing, and resume drilling.
- If a well is to be installed in the borehole, the sandpack and grout are placed as the core-barrel and over-ride casing(s) are selectively vibrated out of the ground. The vibratory action reportedly facilitates the settlement of the sandpack and grout. Upon completion, no casing is left in the ground other than the well casing and screen.

3. Limitations

- Disturbance of the soil core is most likely to occur during removal of the soil core from the core barrel. The soil cores are usually vibrated out of the core barrel into plastic bags approximately 5 feet in length. As the plastic bags are a little larger than the soil core itself, fragmentation of the soil core may occur as the core is extruded into the bag or while the bagged core is being moved in an unsupported manner. Soil conditions that are prone to disturbance include wet or dry zones that contain little or no fines, and well graded sands that contain significant volumes of water.
- If integrity of the soil core is of concern, the following procedures should be implemented:
    i. Measures should be taken to ensure that the core, from the time it is extruded from the core barrel, is rigidly supported through the use of some type of cradle or carrying device.
    ii. The core should not be removed from its cradle until all sampling of the core has been completed. Acrylic liners are available for some core sizes and can be used to hold the core together upon removal from the core barrel
    iii. If the soil is to be sampled for VOCs, acrylic liners must be used.
    iv. Sampling of the soil core for VOCs or SVOCs must be approved on a case by case basis. Proposals for VOC or SVOC soil core sampling must include provisions to minimize core fragmentation and heat generation, such as:
        1. the use of acetate liners in the core barrel so that the soil core does not have to be extruded out of the core barrel;
        2. limiting the length of soil core generated during a given downhole run and;

3. implementing practices to reduce the residency time of the soil core in the core barrel.

- For the analysis of SVOCs, the use of the acetate liners is not required. The large diameter of the core barrel enables ground water sampling equipment to be placed inside the core barrel so that discrete depth groundwater samples can be collected during borehole advancement.

4. References

Standard Practice for Design and Installation of Ground Water Monitoring Wells in Aquifers (October 1990), American Society for Testing and Materials [ASTM] D5092-90.

5. Attachments

None

6. Contact

Melissa Felter

STANDARD OPERATING PROCEDURE

DM-006 GeoProbe ® Direct Push Boring

1. Objective

The purpose of this SOP is to standardize soil sample collection using GeoProbe and MacroCore technologies. A Geoprobe relies on a relatively small amount of static (vehicle) weight combined with percussion as the energy for advancement of a tool string. Using a Geoprobe, you can drive a Macrocore® to obtain continuous soil cores or discrete soil samples.

2. Execution

- Contact the Owner to determine the locations of underground utilities/obstructions. Verify with the contractor that the utility clearance service of the particular state you're working in has been contacted. Ask the subcontractor to provide you with the utility clearance authorization number and the time of clearance to proceed, and record the number in the field notebook (See SOP PM-001).
- Inspect the drilling rig to make sure it is clean and that the down-hole equipment has been steam-cleaned. Check that the steam-cleaner is working properly (i.e., that steam is being produced). Measure and record lengths of all down-hole drilling equipment, including the drilling heads and miscellaneous rods and attachments. Record all observations and measurements in the field notebook.
- Insert a Macrocore (MC) ® liner, (PVC for example) into the sample tube, and connect an MC drive head to the top of the sample tube. A diagram of the MC assembly is provided as Attachment A.
- The drive head is then tightened into the sample tube, and a drive cap is attached to the drive head.
- Place the sampler in the driving position, and drive the sampler until the drive head reaches the ground surface.
- Remove the drive cap, attach a pull cap to the sampler drive head, and pull the sampler out of the ground.
- Remove the cutting shoe and filled liner.
- When the sampler is brought to the ground surface, it should be opened immediately, and the length of recovery should be measured and recorded.
- Decontaminate the sampler if necessary (SOP QA-001) and reassemble the parts with a new liner, and insert the sampler down the same hole to take the next soil core.
- In non-cohesive soils, slough material may enter the sampler as the next core is collected (see limitations below).

- Careful logging of soil stratigraphy is necessary to document whether soil sloughing has occurred within the borehole (see limitations).
- Remove the sample with a clean laboratory spoon and transfer it directly to a suitable sample container.
- Label, preserve, and store the sample in accordance with these Standard Procedures.

3. Limitations

- The GEI oversight person shall ensure that the borehole created by the macro core sampling tube does not collapse between collection of each sample. If the borehole collapses and representative samples cannot be obtained using the standard macro-core sampler, then one of two options may be used.
    i. The macro-core sampler can be fitted with a piston rod assembly, or a 1.5-inch O.D., large bore sampler equipped with a piston rod assembly may be used to collect the samples. The sample tube (macrocore) is advanced through the caved-in borehole material to the top of the desired sampling interval. The sample tube remains closed by a piston tip as it is advanced. Upon reaching the target sample depth, the piston tip will be released and the discrete sampler device is then advanced to collect the representative sample.
    ii. The piston rod assembly is driven up to the top of the sample tube as the sample enters the tube.
- Because the macro-core sampling tube uses a dedicated, disposable liner made of clear plastic, the only part of the sampler that contacts the soil sample is the cutting shoe. Each sample liner will be disposed of after use and a new liner will be placed in the macro-core tube prior to collection of subsequent samples. Cutting shoes and sample collection spoons used to transfer samples to the laboratory jars will be decontaminated between use.

4. References

ASTM D6001-05 Guide for Direct Push Water Sampling for Geoenvironmental Investigations, April 2005.

GeoProbe Systems, "GeoProbe MacroCore MC-5 1.25-inch Light Weight Center Rod Soil Sample System SOP", Technical Bulletin No. MK 3139, November 2006.

5. Attachments

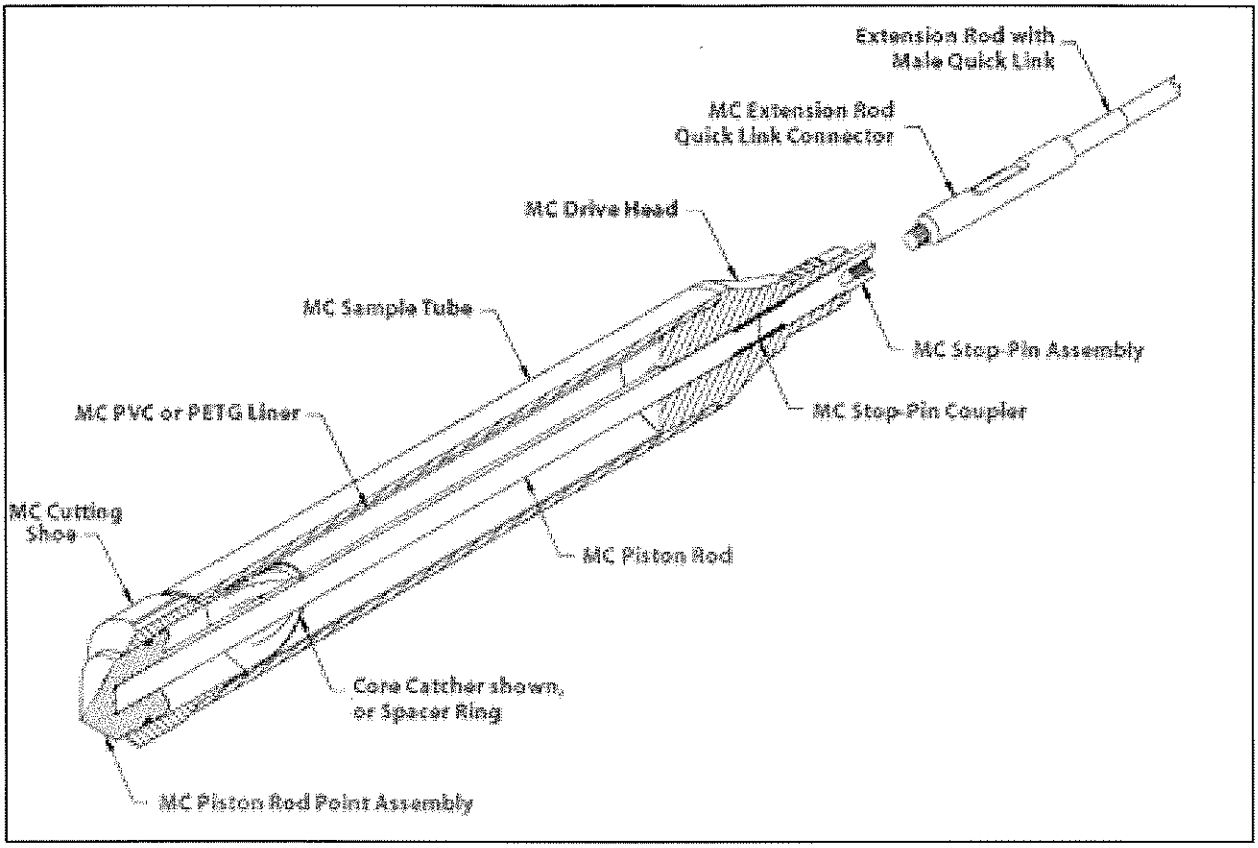
Attachment A – GeoProbe® with Macrocore® Sampler Assembly

6. Contact

Melissa Felter

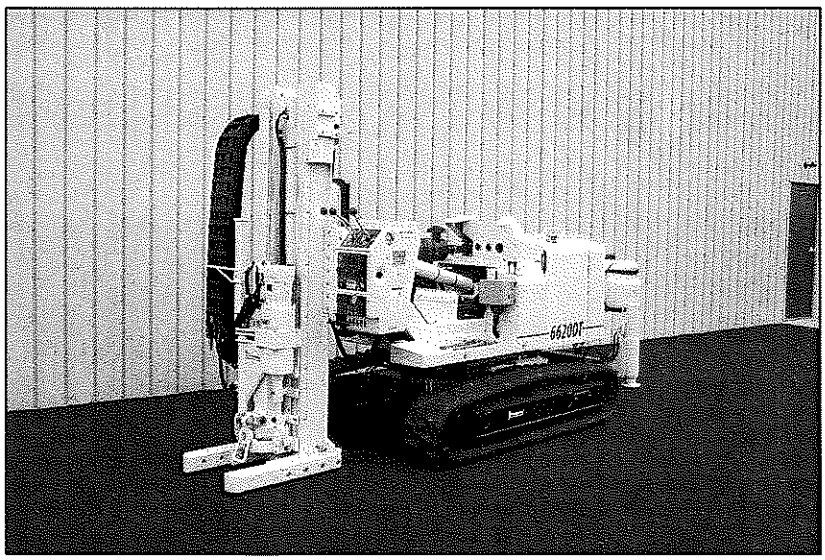
SOP DM-006

Attachment A – GeoProbe® with Macrocore® Sampler Assembly



Above: Diagram of a Macrocore® sampler

Right: A track-mounted GeoProbe ® Rig



GEI Consultants

STANDARD OPERATING PROCEDURE

DM-007 Monitoring Well Construction and Installation

1. Objective

The objective of this SOP is to standardize the installation of shallow overburden monitoring wells for environmental investigations. This SOP addresses the installation of monitoring wells screened across the groundwater table and assumes the monitoring wells will be constructed of flush-joint PVC pipe; the screened section will have factory-slotted openings. Well dimensions (well diameter, screen length, and screen slot-diameters) will be specified in the Work Plan.

2. Execution

- Using a weighted tape, measure and record the depth of the completed soil boring before beginning the well installation.
- Measure the depth to groundwater in the borehole over a 10 to 15-minute period to ensure that the groundwater elevation has approximately stabilized. Compare the saturated soil depth estimated from split-spoon samples to the measured water level in the borehole. If drilling water has been used during boring advancement, pump the water out of the borehole to the static water depth (based on examination of the soil samples) and monitor the recovery of groundwater until the level has stabilized.
- Choose the monitoring well screen and riser lengths so that the slotted section of the screen intersects the groundwater table. If the borehole is deeper than the desired well depth, then fill the base of the borehole with bentonite.
- A minimum of a 4-inch sump should extend to the bottom of the well.
- Monitoring well should be constructed of either 2 or 4 inch ID Schedule 40 threaded-flush jointed PVC.
- Install and secure a bottom well cap. The bottom cap should be secured with either a threaded coupling and/or stainless steel screws.
- Place at least 12 inches of clean uniformly-graded medium quartz filter sand pack into the base of the borehole. Measure and record the depth of the boring. Temporarily cover the top of the riser pipe and lower the complete well plus riser into the borehole, with the base resting on the sand pack.
- Add adequate sand to surround the area around the slotted section. The filter sand should extend at least 2 feet above the top of the slotted section.
- Remove the drilling casing/augers from the borehole slowly, at a maximum of 2-foot intervals. As the drillers pour or use tamping rods to place the filter sand in the borehole, take frequent measurements of

the depth-to-sand. Do not let the sand "bridge" in the annular space. Continue to observe the water level in the borehole.

- Place at least 1 foot of bentonite seal above the filter pack. If the seal is above the water table, use at least 5 gallons of potable water to hydrate the bentonite.
- If necessary, pump bentonite-cement grout using a tremie pipe into the bottom of the annular space to the ground surface. Grout should be mixed in approximately the following proportions: 7.5 gallons water to one 94-lb. bag of cement to 2-4 lbs of pulverized bentonite. The grout must be mixed using the pump on the rig to ensure proper mixing. The protective casing should be set in the grout before it sets.
- The protective surface casing will be either a flush-mounted roadbox or a steel "stick up" pipe. The base of either type of casing must extend at least 1 foot into the grout below the ground surface (below the frost line) whenever possible.
- Cut the monitoring well riser flat and place a mark or "V"-notch or an arrow on the casing with an indelible marker at one point for surveying and groundwater measurements. Cut the well riser so that the top of the well is 3 to 6 inches below the top of the protective casing.
- Set bentonite-cement grout in the annular space between the protective casing and the borehole up to the ground surface. Slope the concrete radially away from the protective casing at the ground surface to promote surface water runoff. In areas of high traffic or areas of parking lots and/or roadways where plowing occurs, set the roadbox FLUSH with the ground surface to avoid damage to the well.
- If the well is installed in a high-traffic area with a guardpipe, additional protection such as steel pole bumpers around the guardpipe may be necessary.
- Place a locking cap on the well pipe.
- All well locations should be photodocumented in accordance with SOP FD-004.
- Label the protective well casing with a paint pen and tape out the location to nearby landmarks so that the well may be located in the future (enter this information in the field notebook). If possible, place a brightly colored stake or other identifier adjacent to the well.
- Develop the well (see SOP DM-009, *Monitoring Well Development*).

3. Limitations

- At all times, follow safety procedures as defined in the site-specific Health and & Safety Plan.
- Site-specific conditions must be evaluated to determine appropriate materials/slot
- The water table will fluctuate seasonally and from year-to-year. Try and estimate the maximum high and low elevations of the water table

from the current water table elevation and the season. Place the 10-foot screen so that at least 2 feet of the screen will extend above the top of the screen when water is at its highest. If very substantial fluctuations in the groundwater table are expected, a 15-foot screen is acceptable.

- Do not screen across different hydrostratigraphic units if possible (for example, outwash sands and till) unless specified in the Work Plan or approved by the Project Manager.
- If the formation is composed of a material that is uniformly coarser than the filter sand, than the grain size of the filter sand must be increased. Consideration should also be given to changing the slot size on the well screen. Differences in average grain size should generally not be greater than a factor of two to four times.
- Do not use borehole/auger cuttings for backfill during monitoring well installation. If the cuttings are suspected to contain contamination which was identified during drilling, cuttings are to be containerized for later characterization and not used for filter pack materials.
- Do not screen across a confining (e.g., silt or clay) layer. Backfill all confining layers with hydrated bentonite or grout.

4. References

Standard Practice for Design and Installation of Ground Water Monitoring Wells in Aquifers (October 1990), American Society for Testing and Materials [ASTM] D5092-90.

Nielsen, D.M. (1993), "Correct Well Design Improves Monitoring," Environmental Protection, July, pp. 38-49.

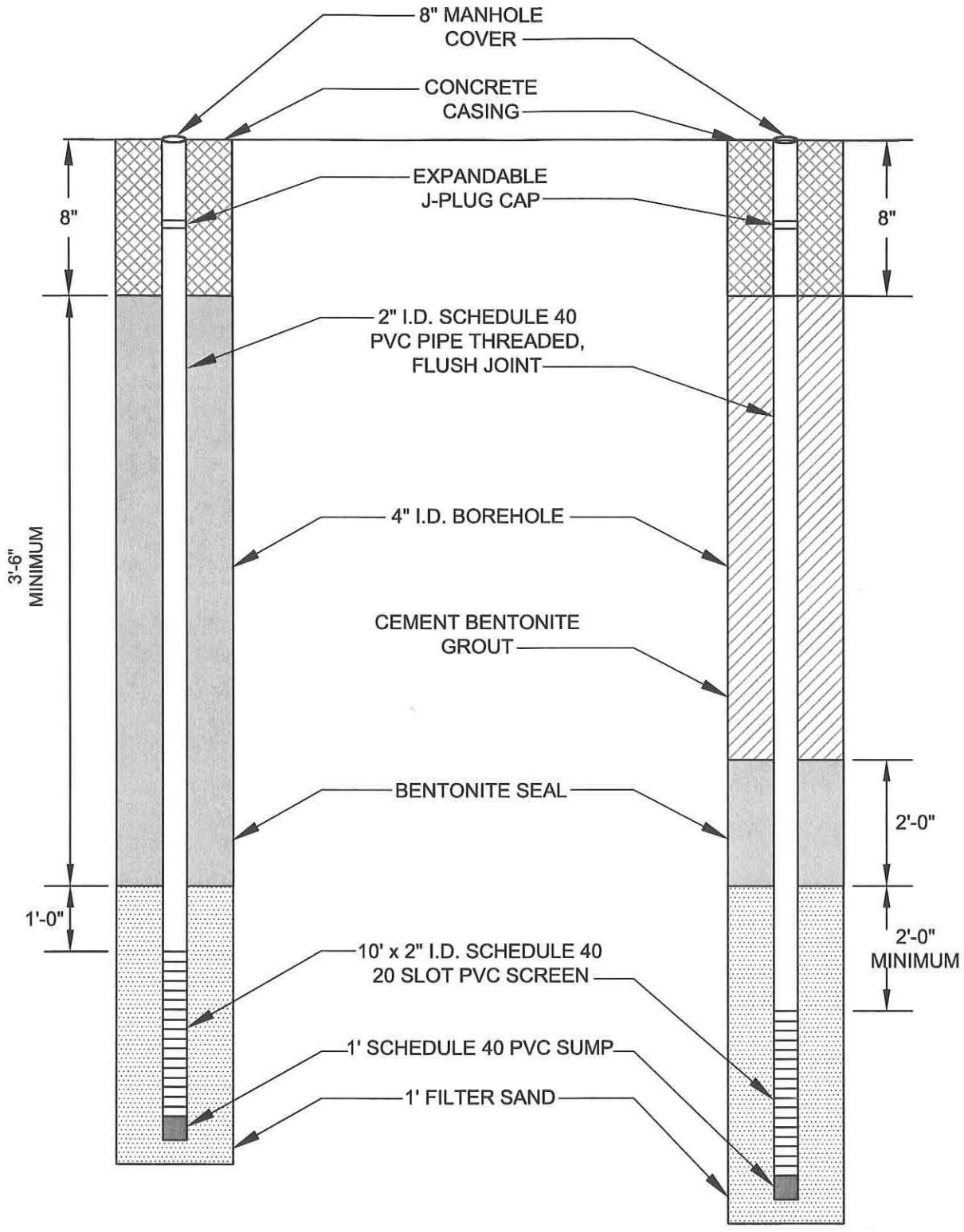
5. Attachments

Attachment A – Typical Shallow, Intermediate, and Deep Groundwater Monitoring Well Construction Detail

6. Contact

David Terry

SHALLOW ***INTERMEDIATE AND DEEP***



NOT TO SCALE

GEI Consultants

TYPICAL SHALLOW, INTERMEDIATE AND DEEP GROUNDWATER MONITORING WELL CONSTRUCTION DETAIL

June 2008

I:\GEI\SOPs\ Well Detail.dwg \Jun 10, 2008

STANDARD OPERATING PROCEDURE

DM-008 Monitoring Well Telescoping

1. Objective

This SOP establishes the method of monitoring well telescoping to prevent the connection of two stratigraphic layers during well installation. Typically, these two stratigraphic layers are overburden and bedrock strata.

2. Execution

- Contact the Owner to determine the locations of underground utilities/obstructions. Verify with the contractor that the utility clearance service of the particular state you're working in has been contacted. Ask the subcontractor to provide you with the utility clearance authorization number and the time of clearance to proceed, and record the number in the field notebook.
- Inspect the drilling rig to make sure it is clean and that the down-hole equipment has been steam-cleaned. Check that the steam-cleaner is working properly (i.e., that steam is being produced). Measure and record lengths of all down-hole drilling equipment, including the drilling heads and miscellaneous rods and attachments. Record all observations and measurements in the field notebook.
- Install large diameter casing (e.g four-inch outer diameter) to the top of the bedrock.
- Drill or core at least ten feet into bedrock to confirm the presence of bedrock and adequately separate stratigraphic units (see precautions below).
- Mix grout. You must have freshly mixed grout continuously to meet requirements. Depending on application a mixture of Portland cement and bentonite meets most grouting requirements. For proper consistency, use no more than 6 gallons of water per 94-pound sack of cement. Add a few pounds of bentonite or hydrated lime per sack of cement for a better flow.
- Use a tremie pipe to deliver grouting outside the casing. This method is not recommended for depths greater than 100 feet. You can use this method if the space between the casing and the borehole wall is large enough to contain a 1-inch tremie pipe. Use the following procedures to complete grouting using this method:
    i. Lower the pipe to the bottom. Make sure that the lower end of the casing is tightly seated at the bottom of the borehole.
    ii. Mix a sufficient quantity of grout and pump it through the tremie pipe or let it descend naturally. As the grout is placed, lift the tremie pipe slowly, but keep the lower end submerged in the grout.

iii. Fill the casing with water as the grout is placed to balance the fluid pressure inside and outside the casing. Doing so prevents grout from leaking under the bottom of the casing.
iv. Allow the grout to set for a minimum of 24 hours.
v. Drill through the existing casing into bedrock to complete monitoring well. Install additional casing, PVC, or open borehole into bedrock.

3. Limitations

- These operating procedures include drilling the borehole used to case off the overburden a minimum of 10 feet into competent bedrock. However, if DNAPL and/or dissolved contamination is suspected or likely to be present in the weathered bedrock, the ten-foot casing requirement will hide the DNAPL from detection. In this case, an overburden well (with casing and screen) should be installed in the weathered bedrock and an outer steel casing installed ten feet into bedrock would not be required.
- If the weathered bedrock is found to be contaminated, a well may need to be installed within the upper 10 feet of competent bedrock. If the well will be constructed with an open hole in the bedrock, an outer steel casing should be installed in the top two feet of competent bedrock to case off the overburden and weathered bedrock aquifers. If casing and screen will be installed in the bedrock aquifer, then installation of the outer steel casing may not be required.

4. References

South Dakota Geologic Survey, "Nine Drilling Methods Standard Operating Procedures Drilling and Monitoring Well Installation at Hazardous Waste Sites in South Dakota SOP 2150", Version 2.0, March 2003.

5. Attachments

None

6. Contact

Gary Fuerstenburg

STANDARD OPERATING PROCEDURE

DM-009 Monitoring Well Development

1. Objective

To remove drilling fluids, to remove fines soil particles that may be trapped in the monitoring well's sand pack and screen, and to set the sand pack so that it will function properly.

2. Execution

- Decontaminate all development equipment prior to use with methanol, Alconox, and deionized-water rinses. See SOP QA-001 Equipment Decontamination.
- Calculate the volume of water in the monitoring well (one well volume).
- Record volume on Monitoring Well Sampling Record (Attachment A).
- Collect a sample of water from the monitoring well with a submersible pump, a bailer, or a Water pump. Record the color and turbidity of the sample. Remove the greater of the following amounts of groundwater:
    i. Ten well volumes.
    ii. The volume of fluid added during drilling.
- Purge groundwater until it runs clear (<50 NTUs).
- Measure the purge rate (gallons per minute) and total volume purged.
- Monitor the groundwater level in the well during development to determine if the pumping rate is sufficient to create a drawdown in the well.
- Collect groundwater samples every few well volumes during the pumping and record the physical properties (color and turbidity).
- Stop pumping when the purge water is relatively clear. Place a surge block in the monitoring well. Slowly move the surge block up and down in the well. Periodically remove the surge block and purge the groundwater until it is relatively clear again. Start at a slow pace and progress to a faster surging action through time.
- Monitor the turbidity and color of the water during this procedure. The well is considered fully developed when all of the following criteria have been met:
    i. The volume of fluid added during drilling has been removed.
    ii. The water removed from the well is relatively free of fine-grained particles.
    iii. Record the volume of water pumped from the well and the physical properties (color, turbidity) of the water.

3. Limitations

- Always remove groundwater with fine particles from the well before surging. The fine particles may be forced into the well screen by the surging action.
- Pump contaminated groundwater into a properly labeled drum.
- Use a bailer to develop monitoring wells that are installed in soils that are composed of fine-grained silts and clays. Pumping and mechanical surging is not recommended because these more vigorous techniques can cause fine particles to clog the filter pack.
- Sampling of groundwater should not occur within two weeks after development.

4. References

Standard Practice for Design and Installation of Ground Water Monitoring Wells in Aquifers (October 1990), American Society for Testing and Materials [ASTM] D5092-90.

Nielsen, D.M. (1993), "Correct Well Design Improves Monitoring," Environmental Protection, July, pp. 38-49.

"The Methods & Mechanics of Well Development, Part 2 of 5," National Drillers Buyers Guide, March 1993, p. 17.

Standard References for Monitoring Wells (April 1991), Commonwealth of Massachusetts Department of Environmental Protection, WSC-310-91.

5. Attachments

Attachment A - Monitoring Well Sampling Record

6. Contact

Mr. Lynn Willey

GEI Consultants

MONITORING WELL SAMPLING RECORD

PID Reading _____ Job Name _____

Job Number _____ By _____ Date _____

Location _____ Measurement Datum _____

Well Number _____

Pre-Development Information Time (start) _____

Water Level _____ Total Depth of Well _____

One Purge Vol _____ Three Well Volume _____

Water Characteristics

Color _____ ___ Clear ___ Cloudy

Odor ___ None ___ Weak ___ Moderate ___ Strong

Any films or immiscible material ___ None

Volume (gal)	Time	pH	Temp (EC)	Spec. Conductance (ΦS/cm)	Turbidity (NTU)	DO Conc. (mg/L)	ORP (mV)	TDS

Total Volume Removed (gal) _____ pH _____

Temperature (EC) _____ Specific Conductance (ΦS/cm) _____

DO Concentration (mg/L) _____ ORP (mV) _____

_____ TDS _____

Post Development Information Time (Finished) _____

Water Level _____ Total Depth of Well _____

Approximate Volume Removed (gal) _____

Water Characteristics

Color _____ ___ Clear ___ Cloudy

Odor ___ None ___ Weak ___ Moderate ___ Strong

Any films or immiscible material ___ None

_____

Comments:

SUMMARY GUIDANCE

General Guidance on Sample Collection

1. Overview

The primary objective of all sampling activities is to characterize a site accurately so that its impact on human health and the environment can be properly evaluated. It is only through sampling and analysis that site hazards can be measured and the job of cleanup and restoration can be accomplished effectively with minimal risk. The sampling itself must be conducted so that every sample collected retains its original physical form and chemical composition. In this way, sample integrity is insured, quality assurance standards are maintained, and the sample can accurately represent the larger body of material under investigation. The extent to which valid inferences can be drawn from a sample depends on the degree to which the sampling effort conforms to the project's objectives. For example, as few as one sample may produce adequate, technically valid data to address the project's objectives. Meeting the project's objectives requires thorough planning of sampling activities, and implementation of the most appropriate sampling and analytical procedures.

2. Types of Samples

In relation to the media to be sampled, two basic types of samples can be considered:

2.1. Hazardous sample

Hazardous or concentrated samples are those collected from drums, tanks, lagoons, pits, waste piles, fresh spills, or areas previously identified as contaminated, and require special handling procedures because of their potential toxicity or hazard. These samples can be further subdivided based on their degree of hazard; however, care should be taken when handling and shipping any wastes believed to be concentrated regardless of the degree.

2.2. Environmental sample

Environmental samples are those collected from streams, ponds, lakes, wells, and are off-site samples that are not expected to be contaminated with hazardous materials. They usually do not require the special handling procedures typically used for concentrated wastes. However, in certain instances, environmental samples can contain elevated concentrations of pollutants and in such cases would have to be handled as hazardous samples.

The importance of making the distinction between environmental and hazardous samples is two-fold:

- Personnel safety requirements: Any sample thought to contain enough hazardous materials to pose a safety threat should be designated as hazardous and handled in a manner which ensures the safety of both field and laboratory personnel.
- Transportation requirements: Hazardous samples must be packaged, labeled, and shipped according to the International Air Transport Association (IATA) Dangerous Goods Regulations or Department of Transportation (DOT) regulations and U.S. EPA guidelines.

3. Field Screening

The main advantage of field analysis is that it allows for the performance of rapid characterization with only a few mobilizations via a dynamic sampling plan. An unique advantage that is offered by field analysis is dedicated analysis of the field samples with the associated QC samples. Often in a batch of 20 samples in a laboratory, a small number of samples (e.g. three samples) from the site of interest are processed with other unrelated samples and the QC samples (e.g. matrix spike samples) may not be one of the site samples. Therefore, the DQO that requires the quality assurance project plan (QAPP) to be based on the very specific needs of each site is served well or often better by dedicated field analysis. Also, due to a rapid turn-around time, the sample integrity of a properly collected and stored one-hour-old sample is often better than that of a sample held for 14 days.

To be "effective," the field data generated must be of sufficient quality, with respect to measurement precision or reproducibility, accuracy, sensitivity, and have good correlation with the standard laboratory methods to support the objective of the site investigation or cleanup and the DQO. Several factors to be considered before mobilization include the following:

- The action levels for field decisions shall be established as part of the DQOs.
- The project objective shall permit screening and semi-quantitative data in addition to quantitative data to meet DQO.
- The percentage of samples to be analyzed in the field as well as sent off-site for laboratory confirmation shall be determined.
- The methodology to compare field and laboratory data shall be established, for example using duplicate (field duplicate samples) and/or performance evaluation samples in addition to initial and daily calibrations.
- For the field instrument or the analytical method, the measurement selectivity, sensitivity, precision, accuracy, representativeness and action levels shall be determined.
- The standard operating procedures and method detection limit studies are completed before mobilization.
- Evaluate matrix interferences that might be associated with a particular field technology.

- If applicable, the field technician performing the analyses shall have proof of training by the manufacturer/vendor of the test method.
- If sample preservation is required, samples shall be preserved in the field immediately after collection according to the method specific table in chapter two of this document.

4. Sample Collection Techniques

In general, two basic types of sample collection techniques are recognized, both of which can be used for either environmental or hazardous samples.

4.1. Grab Samples

A grab sample is defined as a discrete aliquot representative of a specific location at a given point in time. The sample is collected all at once at one particular point in the sample medium. The representativeness of such samples is defined by the nature of the materials being sampled. In general, as sources vary over time and distance, the representativeness of grab samples will decrease.

4.2. Composite Samples

Composites are non-discrete samples composed of more than one specific aliquot collected at various sampling locations and/or different points in time. Analysis of this type of sample produces an average value and can in certain instances be used as an alternative to analyzing a number of individual grab samples and calculating an average value. It should be noted, however, that compositing can mask problems by diluting isolated concentrations of some hazardous compounds below detection limits. Compositing is often used for environmental samples and may be used for hazardous samples under certain conditions. For example, compositing of hazardous waste is often performed after compatibility tests have been completed to determine an average value over a number of different locations (group of drums). This procedure generates data that can be useful by providing an average concentration within a number of units, can serve to keep analytical costs down, and can provide information useful to transporters and waste disposal operations.

For sampling situations involving hazardous wastes, grab sampling techniques are generally preferred because grab sampling minimizes the amount of time sampling personnel must be in contact with the wastes, reduces risks associated with compositing unknowns, and eliminates chemical changes that might occur due to compositing.

4.3. Types of Sampling Strategies

The number of samples that should be collected and analyzed depends on the objective of the investigation. There are three basic sampling strategies: random, systematic, and judgmental sampling.

- Random sampling involves collection of samples in a nonsystematic fashion from the entire site or a specific portion of a site.
- Systematic sampling involves collection of samples based on a grid or a pattern which has been previously established.
- When judgmental sampling is performed, samples are collected only from the portions) of the site most likely to be contaminated.

Often, a combination of these strategies is the best approach depending on the type of the suspected/known contamination, the uniformity and size of the site, the level/type of information desired, etc.

5. Sample Hold Time, Container, and Preservation Methods

The following table provides required Holding Time, Container, and Preservation Methods. Most of the information is specific to the EPA analytical method and should be pertinent to all sampling schemes. However, some analytical preservation and analytical methods are state specific. Quality assurance plans should clearly identify preservation methods and hold times prior to sampling.

Aqueous

Parameter	Holding Time	Container	Volume	Preservative
Acidity	14 days	P, G	100 ml	Cool, 4°C
Alkalinity	14 days	P, G	100 ml	Cool, 4°C
Biological Oxygen Demand (BOD)	48 hours	P, G	1000 ml	Cool, 4°C
Chemical Oxygen Demand (COD)	28 days	P, G	100 ml	Cool, 4°C, H2SO4 to pH<2
Chloride	28 days	P, G	100 ml	Cool, 4°C
Chromium, Hexavalent	24 hours	P, G	250 ml	Cool, 4°C
Cyanide				
Amenable	14 days1	P, G	500 ml	Cool, 4°C, NaOH to pH>12
Free	14 days1	P, G	500 ml	Cool, 4°C, NaOH to pH>12
Total	14 days1	P, G	500 ml	Cool, 4°C, NaOH to pH>12
Fluoride	28 days	P	100 ml	Cool, 4°C
Hardness, Total	6 months	P, G	100 ml	HNO3 to pH<2
Metals (except Cr+6, Hg)	6 months	P	500 ml	Cool, 4°C, HNO3 to pH<2
MBAS	48 hours	G	500 ml	Cool, 4°C
Mercury	28 days	P, G	500 ml	HNO3 to pH<2
N, Ammonia	28 days	P, G	100 ml	H2SO4 to pH<2
N, T. Kjeldahl	28 days	P, G	500 ml	H2SO4 to pH<2
N, Nitrate	48 hrs/28 days preserved	P, G	100 ml	Cool, 4°C or add H2SO4 to pH<2
N, Nitrite	48 hours	P, G	100 ml	Cool, 4°C
Oil and Grease	28 days	G	1000 ml	Cool, 4°C, H2SO4or HCl to pH<2
Petroleum Hydrocarbons	14 days	G	1000 ml	Cool, 4°C, H2SO4 to pH<2
pH	Analyze Immediately	P, G	50 ml	N/A
Phenols,Recoverable	28 days	G	500 ml	Cool, 4°C, H2SO4 to pH<2
Phosphorus, Ortho	48 hours	P, G	100 ml	Filter, Cool, 4°C
Phosphorus, Total	28 days	P, G	100 ml	Cool, 4°C, H2SO4 to pH<2
Radiological Tests				
Alpha, Beta & Radium	6 months	P, G	4 L	Cool, 4°C, HNO3 to pH<2
Solids, Total	7 days	P, G	100 ml	Cool, 4°C
Solids, Total Dissolved	7 days	P, G	100 ml	Cool, 4°C
Solids, Total Suspended	7 days	P, G	100 ml	Cool, 4°C

Aqueous (cont.)

Solids, Volatile Suspended	7 days	P, G	100 ml	Cool, 4°C
Sulfate	28 days	P, G	100 ml	Cool, 4°C
Total Organic Carbon	28 days	P, G	100 ml	Cool, 4°C, H2SO4 to pH<2
Halogenated Volatiles	14 days	40 ml vials	2x40 ml	Cool, 4°C3
Purgeable Aromatics	14 days[4]	40 ml vials	2x40 ml	Cool, 4°C, HCl to pH<2
Phenols by GC/MS	7 days/40 days[5]	G	1 L	Cool, 4°C
Pesticides/PCBs	7 days/40 days[5]	G	1 L	Cool, 4°C
Polynuclear Aromatics	7 days/40 days[5]	G	1 L	Cool, 4°C
Acid/Base-Neutral Extractables	7 days/40 days[5]	G	1 L	Cool, 4°C

Solid

Parameter	Holding Time	Container	Volume	Preservative
Metals (except Hg)	6 months	P, G	100 g	Cool, 4°C
Mercury	28 days	P, G	100 g	Cool, 4°C
Halogenated Volatile Organics	14 days	G	10 g/10 ml methanol	Methanol preserved in field[6]
Purgeable Aromatics	14 days	G	10 g/10 ml methanol	Methanol preserved in field[6]
Phenols	14 days/40 days[5]	G	100 g	Cool, 4°C
Pesticides/PCBs	14 days/40 days[5]	G	100 g	Cool, 4°C
Polynuclear Aromatics	14 days/40 days[5]	G	100 g	Cool, 4°C
Acid/Base-Neutral Extractables	14 days/40 days[5]	G	100 g	Cool, 4°C

P = Plastic G = Glass

[1]If residual chlorine is present, add 0.6 gm. ascorbic acid.

[2]Maximum holding time is 24 hours when sulfide is present. Test with lead acetate paper prior to pH adjustment. Remove sulfide with addition of lead nitrate until a negative spot test is obtained. Filter and add NaOH to pH>12.

[3]If samples contain residual chlorine, add 0.008% sodium thiosulfate at the time of sampling.

[4]With pH adjustment; without, holding time is 7 days.

[5]Seven days prior to extraction. Samples must be analyzed within 40 days after extraction.

[6]Encore samplers may be used, but must be received in lab and extracted within 48 hours.

STANDARD OPERATING PROCEDURE

SC-002 Sample Handling

1. Objective

Sampling handling involves the collection and shipping of environmental samples to a laboratory for chemical analysis. The overall objective of sample handling is to ensure that samples are properly:

- labeled and documented;
- preserved;
- packaged; and
- transported to laboratories.

2. Execution

- Prior to mobilizing to the field, select a shipper or arrange for a courier for sample delivery to the laboratory. If using a shipper (i.e., Federal Express, UPS) determine the time constraints for pickup requests, the location and hours of the nearest shipping office, and any size/weight restrictions.
- Label all laboratory glassware with waterproof ink prior to collecting samples. The label should have an adhesive and be placed on the jar or bottle, not on the cap.
- Record the following information on the label and in the field notebook (See Field Notebook SOP FD-001): project number, sample identification (i.e., MW201 or SS-2), date and time (military time) of collection, sampler's initials, and preservative, if present.
- If sample jars are not prepreserved, add preservative as appropriate.
- At each sampling location, samples must be collected in order of volatility, most volatile first. Samples collected for volatile analysis must be placed in sample containers immediately upon retrieval of the sample.
- Aqueous samples for volatile analysis must be collected without air bubbles. Soil samples for volatile analysis should be compacted to eliminate as much headspace as possible. Other laboratory glassware should also be filled when possible. Care must be taken to avoid getting soils on the threads of sample jars, which can cause a faulty seal.
- If compositing of samples is performed in the field, specify basis for composite (i.e. volume, weight, spoon recovery, etc.) and record procedure for compositing sample in the field book.
- Once samples have been collected, place samples in a cooler with ice or a blue pack and start the chain-of-custody form (SOP FD-003 Sample Handling and *Chain-of-Custody*).

- For shipping, individually wrap each sample bottle with bubble packing@ or suitable packing material and place the wrapped bottles in the cooler with sufficient packing material between samples to avoid breakage.
- Place a layer of packing material above and below the sample bottles. Place blue ice packs or ice bags on top of the packing material. Fill the remaining space in the cooler with packing material to eliminate the possibility of vertical movement of samples.
- Place the completed and signed chain-of-custody form in a plastic bag and place on top of the packing material in the cooler.
- Fill out the appropriate shipping or courier forms and attach to the top of the cooler. If necessary, place the proper shipping labels on the cooler. Have the courier sign the chain-of- custody form (or write pickup by FEDEX, UPS, etc. with date and time). Place a custody seal on the cooler.
- A copy of the waybills must be kept by the field supervisor to trace shipments if necessary.

3. Limitations

- At all times, follow safety procedures as defined in the site-specific Health and Safety Plan.
- Field personnel must be aware of analyses which have short holding times and schedule sampling events and shipping accordingly. Shipment of samples for analyses with short holding times must be planned in advance. Refer to the project work plan, quality assurance project plan, or state/federal regulations for holding time and preservative information.
- In general, glassware for aqueous samples contains preservatives, i.e.,HNO3, HCl, etc. When collecting the sample, take care not to overfill the container, thus flushing the preservative out of the bottle.
- Never composite samples for VOCs in the field. Collect individual aliquots and direct the laboratory to perform compositing.
- Collection of aqueous samples should not be performed over the opening of a monitoring well. Preservatives from overfilling, a marker pen or other objects could fall into the well.
- If the recharge volume for a monitoring well is low, completely fill all volatile vials and then collect the minimum sample volume required for each remaining analysis.
- During subsurface soil sampling, if the recovery from the split-spoon sample is inadequate, if appropriate, resample the bottom of the borehole to obtain proper sample volume.

- Laboratories will homogenize and test the contents of the sample container, unless directed otherwise. Samples should not contain rocks, twigs, leaves, etc. unless these materials are of interest.

4. References

New Jersey Department of Environmental Protection, Field Sampling Procedures Manual, August 2005.

Connecticut Department of Environmental Protection, Guidance for Collecting and Preserving Soil and Sediment Samples for Laboratory

Determination of Volatile Organic Compounds, Version 2.0 February 28, 2006.

5. Attachments

None

6. Contact

Lynn Willey

STANDARD OPERATING PROCEDURE

SC-003 Investigation Derived Waste (IDW)

1. Objective

The objective of this SOP is to provide guidelines for the proper management of investigational Derived Waste (IDW) resulting from site investigation activities. This SOP addresses IDW generated during field tasks typically performed for environmental site investigations. The intent of this SOP is to provide a set of guidelines for proper assessment and handling of these IDWs.

2. Execution

- Determine the suspected contamination type and impacted media anticipated, based on previous investigations, current analytical data, and/or site history.
- Consider the following issues when selecting IDW management option(s):
    i. anticipated volume of IDW to be generated during on-site activities
    ii. potential contaminants and their concentrations
    iii. location of the nearest populations and the likelihood and/or degree of site access
    iv. potential exposures to workers
    v. potential for environmental impacts
    vi. community concerns
    vii. potential storage areas
    viii. regulatory constraints
    ix. potential on-site treatment options
- Review IDW Management Options summarized in Attachment A for each media suspected of contamination.
- Select IDW Management Option(s) prior to the commencement of field activities that will generate waste materials.
- Include the selected IDW Management Option(s) in the Field Plan.
- In addition to the issues considered above for the selection of IDW management strategies/disposal options, more specific considerations/guidelines include:

2.1. Test Pit Excavation

- Segregate contaminated soil from uncontaminated soil using visual and/or field screening methods.
- Use appropriate barrier (two layers of 6-ml plastic sheeting) for temporary stockpiling of contaminated soil adjacent to test pit.
- Backfilling of test pits with contaminated soil.

- For situations where returning contaminated soil to the test pit is deemed protective by the project manager, backfill soil in the same order as the soil was excavated from the test pit.

2.2. Boring/Monitoring Well Installation

- For auger borings, segregate contaminated soil (determined by visual and/or field screening methods) from uncontaminated soil during drilling. Segregate residual contaminated soil from split-spoon sampling.
- Auger cuttings or sediment generated by drive and wash may be spread around the ground surface at the boring location if deemed appropriate by the project manager. IDW may be placed in an appropriate area or container pending characterization and appropriate disposal. (A useful rule of thumb is to assume generation of one 55-gallon drum of cuttings for each 20 feet drilled with 7-¼-inch-I.D. augers).
- Segregate contaminated drilling fluid from uncontaminated fluid for rotary wash borings.
- Drilling fluid management options include pouring the drilling fluid on the ground in the Area of Concern (AOC) or containerizing the fluid in drums or tanks.

2.3. Water Development/Sampling

- Contaminated groundwater removed from wells by pumping or bailing for the purpose of well development and sampling may be poured on the ground in the AOC or containerized in drums or tanks at the project manager's discretion.

2.4. Decontamination Fluids

- Decontamination fluids may only be poured on the ground in the vicinity of the well in situations deemed protective by the project manager. Alternatively, the fluids may be containerized in drums or tanks.

2.5. Disposable Personal Protective Equipment

- Disposable PPE must be managed like any other IDW. It should only be removed from the site with the project manager's approval, and may be disposed of as ordinary rubbish only if it has not come into contact with hazardous materials.

3. Limitations

- The preferred IDW management option is to return the IDW to its source.

- The IDW selected must be in accordance with state/federal regulations.
- Our clients are responsible for the disposal of IDW, should disposal be necessary.

4. References

Guide to Management of Investigation - Derived Wastes (April 1992), United States Environmental Protection Agency, Publication 9345.3-03FS.

Standard References for Monitoring Wells, Massachusetts Department of Environmental Protection, Publication No. WSC-310-91.

5. Attachments

Attachment A: Summary of IDW Management Options

6. Contact

David Terry

Table 1: Summary of IDW Management Options

Attachment A: - SUMMARY OF IDW MANAGEMENT OPTIONS
GEI Consultants, Inc. Standard Operating Procedures
Management of Investigation - Derived Waste

Type of IDW	Generation Processes	Management Options	Remarks
Soil	Boring/monitoring well installation Test pit excavation Soil sampling	Return to source immediately after generation	OK only if deemed "protective" by project manager and regulatory agency
		Spread around boring test pit or source within the AOC	OK only if deemed "protective" by project manager and regulatory agency
		Send to on-site TDU within AOC	Need Interim or Permanent permit if on-site storage >90 days
		Send to off-site TDU within 90 days	Requires manifests, analytical characterization
		Store for future treatment and/or disposal. Storage consistent with state/federal regulations.	Storage container/pile/tank in accordance with 40 CFR Part 264 and 265 (see notes)
		Store temporarily awaiting laboratory analysis. Storage consistent with state/federal regulations.	
Sediment/Sludge	Sludge pit sampling Sediment sampling	Return to source immediately after generation	OK only if deemed "protective" by project manager and regulatory agency
		Send to on-site TDU within AOC	Need Interim or Permanent permit if on-site storage >90 days
		Send to off-site TDU within 90 days	Requires manifests, analytical characterization
		Store for future treatment and/or disposal. Storage consistent with state/federal regulations.	Storage container/pile/tank in accordance with 40CFR Part 264 and 265

Attachment A: - SUMMARY OF IDW MANAGEMENT OPTIONS
GEI Consultants, Inc. Standard Operating Procedures
Management of Investigation - Derived Waste

Type of IDW	Generation Processes	Management Options	Remarks
Aqueous liquids (groundwater, surface water, drilling fluids, other wastewater)	Well installation/development Well purging during sampling Ground water discharge - pump tests Surface water sampling	Pour onto ground close to well (nonhazardous)	Ensure that it is permissible by local, state, and Federal regulations
		Send to on-site TDU within AOC	Storage in accordance with 40 CFR Part 264 and 265, 90-day limit
		Send to off-site commercial treatment unit within 90 days	Requires manifests, analytical characterization
		Send to POTW	Obtain appropriate discharge permit(s)
		Store for future treatment and/or disposal. Storage consistent with state/federal regulations.	Consistent w/ final remedial action
		Discharge to surface water	OK only if deemed "protective," complies w/ CWA, USCG regulations. Obtain appropriate discharge permit(s).

Decontamination fluids	Decon of PPE and equipment	Send to on-site TDU within AOC	Storage in accordance with 40 CFR Part 264 and 265, 90-day limit
		Send to off-site TDU within 90 days	Requires manifests, analytical characterization
		Store for future treatment and/or disposal. Storage consistent with state/federal regulations.	Consistent with final remedial action
Disposable PPE	Sampling, drilling, and test pit excavation observation, other on-site activities	Send to on-site TDU within AOC	Dispose of appropriately after characterization
		Place in on-site industrial dumpster	Project-specific determination required
		Send to off-site TDU within 90 days	Project-specific determination required
		Store for future treatment and disposal. Storage consistent with state/federal regulations.	Project-specific determination required

Notes:

1) AOC - area of contamination
2) TDU - temporary disposal unit
3) PPE - personal protective equipment
4) POTW - publicly owned treatment works
5) Generation processes listed here are provided as examples. IDW may also be generated as a result of other site activities.
6) RCs - Reportable Concentrations
7) RCRA Container/Waste Pile/Tank requirements:
   Containers; 40 CFR 264 Subpart I and 265 Subpart I
   Waste Piles; 40 CFR 264 Subpart L and 265 Subpart L
   Tanks; 40 CFR 264 Subpart J and 265 Subpart J

STANDARD OPERATING PROCEDURE

SC-004 Head Space VOC Screening

1. Objective

To obtain a site-specific measurement of the VOC concentrations present in soil. This information can be used: 1) to segregate soil based on degree of contamination, 2) to identify samples for quantitative analysis of VOCs, or 3) as a qualitative method to evaluate the presence or absence of VOCs in soil.

2. Execution

- A photoionization detector (PID) or flame ionization detector (FID) instrument is used to measure VOCs in jar head space (JHS) screening. Select the appropriate instrument, lamp, and calibration gas for the site-specific contaminants. Calibrate the instrument in accordance with the manufacturer's instructions before JHS screening begins. Record the type of calibration gas, detector, and lamp in the field notebook.
- Note the highest VOC concentration that the instrument measures in air in the work area before performing JHS screening. Record this as the initial background concentration.
- Half-fill a clean, glass jar with the soil. Use a clean trowel or soil spatula. Quickly cover the open top with one or two sheets of clean, aluminum foil and screw on the cap to tightly seal the jar. Label the sample location and depth from which the sample was collected on the jar.
- Allow headspace development for at least 10 minutes at an ambient temperature of 50°F or greater. Vigorously shake the jar for 15 seconds at the beginning and end of the headspace development period. When ambient temperatures are below 50°F, place the jar in a heated van or building during the headspace development period.
- After headspace development, remove the screw cap to expose the foil seal. Quickly puncture the foil seal with the instrument's sampling probe and insert it to a point at about one-half of the headspace depth.
- Record the highest VOC concentration that the instrument displays as the JHS concentration. The highest concentration should occur between 2 and 5 seconds after probe insertion.

3. Limitations

- The instruments may work poorly in the rain and in freezing temperatures. Under such conditions, operate the instrument in a heated vehicle or building.
- Prevent water and soil particles from entering the tip of the instrument probe. Use a filter on the instrument's probe.

- Measure background VOC conditions and perform JHS screening away from non-site-related VOC sources, such as vehicle and heavy equipment exhaust.
- The VOC concentration on the instrument's display may vary when the instrument measures when the air contains high VOC concentrations or high moisture.
- JHS screening is a guide that helps the screener to segregate soils into broadly defined categories. JHS screening results may differ by orders of magnitude from laboratory testing results.
- Note that states may have specific procedures for field monitoring. In Massachusetts, the Massachusetts Department of Environmental Protection (DEP) requires that screening of gasoline-contaminated soil be performed in accordance with Attachment II of the DEP's policy #WSC-94-400 Interim Remediation Waste Management Policy for Petroleum Contaminated Soils. Under this policy, two samples need to be taken at each sampling point and compared; replicate values should be consistent to plus or minus 20%. The instrument should be calibrated to read ppm as benzene with a 10.0 (+/-) eV lamp source. Instrument calibration should be checked/adjusted once every 10 samples, or daily, whichever is greater.

4. References

Interim Remediation Waste Management Policy for Petroleum Contaminated Soils. (April 1994), Massachusetts Department of Environmental Protection, Policy #WSC-94-400.

5. Attachments

None

6. Contact

Mr. Lynn Willey

STANDARD OPERATING PROCEDURE

SM-001 – Soil Sampling Techniques

1. Objective

This SOP is used primarily to collect surface and shallow subsurface soil samples. Surface soils are generally classified as soils between the ground surface and 6 to 12 inches below ground surface. The shallow subsurface interval may be considered to extend from approximately 12 inches below ground surface to a site-specific depth at which sample collection using manual methods becomes impractical. Refer to state-specific regulations as to the exact definition of what constitutes a surface soil.

2. Execution

2.1. Surface Soil Sampling

Collection of samples from near-surface soil can be accomplished with tools such as spades, shovels, trowels, and scoops. Surface material is removed to the required depth and a stainless steel or plastic scoop is then used to collect the sample. This method can be used in most soil types but is limited to sampling at or near the ground surface. Accurate, representative samples can be collected with this procedure depending on the care and precision demonstrated by the sample team member. A flat, pointed mason trowel to cut a block of the desired soil is helpful when undisturbed profiles are required.

- Carefully remove the top layer of soil or debris to the desired sample depth with a pre-cleaned spade.
- Using a pre-cleaned, stainless steel scoop, plastic spoon, or trowel, remove and discard a thin layer of soil from the area which came in contact with the spade.
- If volatile organic analysis is to be performed, transfer the sample directly into an appropriate labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly.
- Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval.
- Either place the sample into appropriate labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval or location into the homogenization container and mix thoroughly.
- When compositing is complete, place the sample into appropriate labeled containers and secure the caps tightly.

2.2. Sampling at Depth with Augers and Thin Wall Tube Samplers

This system consists of an auger, or a thin-wall tube sampler, a series of extensions, and a "T" handle. Attachment A provides a diagram of these items. The auger is used to bore a hole to a desired sampling depth, and is then withdrawn. The sample may be collected directly from the auger. If a core sample is to be collected, the auger tip is then replaced with a thin wall tube sampler. The system is then lowered down the borehole, and driven into the soil to the completion depth. The system is withdrawn and the core is collected from the thin wall tube sampler. Several types of augers are available; these include: bucket type, continuous flight (screw), and post-hole augers. Bucket type augers are better for direct sample recovery because they provide a large volume of sample in a short time. When continuous flight augers are used, the sample can be collected directly from the flights. The continuous flight augers are satisfactory when a composite of the complete soil column is desired. Post-hole augers have limited utility for sample collection as they are designed to cut through fibrous, rooted, swampy soil and cannot be used below a depth of approximately three feet.

The following procedure is used for collecting soil samples at depth with the auger:

- Attach the pre-decontaminated auger bit to a drill rod extension, and attach the "T" handle to the drill rod.
- Clear the area to be sampled of any surface debris (e.g., twigs, rocks, litter). It may be advisable to remove the first three to six inches of surface soil for an area approximately six inches in radius around the drilling location.
- Begin augering, periodically removing and depositing accumulated soils onto a plastic sheet spread near the hole. This prevents accidental brushing of loose material back down the borehole when removing the auger or adding drill rods. It also facilitates refilling the hole, and avoids possible contamination of the surrounding area.
- After reaching the desired depth, slowly and carefully remove the auger from the hole. When sampling directly from the auger, collect the sample after the auger is removed from the hole.

2.3. Thin- Walled Core Sample

- Remove auger tip from the extension rods and replace with a pre-cleaned thin wall tube sampler. Install the proper cutting tip.
- Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into the soil. Do not scrape the borehole sides. Avoid hammering the rods as the vibrations may cause the boring walls to collapse.

- Remove the tube sampler, and unscrew the drill rods.
- Remove the cutting tip and the core from the device.
- Discard the top of the core (approximately 1 inch), as this possibly represents material collected before penetration of the layer of concern. Place the remaining core into the appropriate labeled sample container. Sample homogenization is not required.
- If volatile organic analysis is to be performed, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly.
- When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.
- If another sample is to be collected in the same hole, but at a greater depth, reattach the auger bit to the drill and assembly, and follow previous steps, making sure to decontaminate the auger and tube sampler between samples.
- Abandon the hole according to applicable state regulations. Generally, shallow holes can simply be backfilled with the removed soil material.

2.4. Sampling at Depth with a Split Spoon (Barrel) Sampler

Split spoon sampling is generally used to collect undisturbed soil cores of 18 or 24 inches in length. A series of consecutive cores may be extracted with a split spoon sampler to give a complete soil column profile, or an auger may be used to drill down to the desired depth for sampling. The split spoon is then driven to its sampling depth through the bottom of the augured hole and the core extracted. A diagram of the split-spoon sampler assembly is provided as Attachment A.

When split spoon sampling is performed to gain geologic information, all work should be performed in accordance with ASTM D1585-98, "Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils". The following procedures are used for collecting soil samples with a split spoon:

- Select the size (length and diameter) of split-spoon sampler based on the amount of soil that is needed for characterization. The ASTM standard for N-values is 1 3/8 - inch I.D. Specify spoon size and basket type to driller prior to mobilization to the site.

- Select a soft or stiff basket for the spoon (a softer basket works better for loose or soft material).
- Prior to hammering the split spoon to collect the sample, verify that the split-spoon is no more than 6 inches above the top of the desired sample depth. If the split spoon is more than 6 inches above the top of the desired sample depth, clean out the hole prior to sampling. Record all depth measurements relative to ground surface.
- Assemble the sampler by aligning both sides of barrel and then screwing the drive shoe on the bottom and the head piece on top. See diagram in Attachment A.
- Place the sampler in a perpendicular position on the sample material.
- For all soil samples, use a 140-lb hammer to drive the sampler, unless conditions necessitate using a 300-lb hammer.
- Record in the site logbook or on field data sheets the length of the tube used to penetrate the material being sampled, and the number of blows required to obtain this depth.
- Count and record the number of blow counts per 6-inch increments (confirm blow counts with driller if necessary).
- Withdraw the sampler, and open by unscrewing the bit and head and splitting the barrel. The amount of recovery and soil type should be recorded on the boring log. If a split sample is desired, a cleaned, stainless steel knife should be used to divide the tube contents in half, longitudinally. This sampler is typically available in 2 and 3 1/2 inch diameters. A larger barrel may be necessary to obtain the required sample volume.
- Without disturbing the core, transfer it to appropriate labeled sample container(s) and seal tightly.
- Note any material in the nose (shoe) of the spoon.
- Immediately collect a sample for VOCs (if required by the site-specific field sampling plan) by collecting soil from the entire length of the split spoon, unless otherwise specified. When the most impacted interval (based on field instrument screening) is sampled for laboratory analysis, screen the spoon with the field instrument first, then collect the soil sample for VOC analysis from the appropriate interval.
- Screen the soil sample for VOCs or other constituents as indicated in the site-specific field sampling plan.

3. Limitations

- At all times, follow safety procedures as defined in the Site-Specific Health and Safety Plan.
- Weather conditions (e.g. frozen ground) may prevent the collection of samples and should be considered prior to sample collection.
- Tools plated with chrome or other materials should not be used.

3.1. Split Spoon Sampling

- Be aware of the length of the drill string, the sample depth, and the required, stickup of the drill string to ensure accurate sample interval measurement.
- If drilling with hollow stem augers, the removal of the drill string from the hole, prior to attaching the split-spoon sampler, may cause soils to be sucked up into the augers (blow-in running sands). Upon recovery, determine what is blow-in. In general, blow-in is more unconsolidated than the rest of the sample and lacks stratification (do not include blow-in for recovery of sample collection).
- If soils are loose sands or soft clay, the drill string and sampler may advance slightly under its own weight, giving a false depth for soil collection.
- Never sample more than two spoons consecutively unless material is tight. Do not let the split spoon penetrate more than it can hold.
- In many instances groundwater will fill the auger and the split sppon

4. References

ASTM D1585-98, "Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils". 1998

United States Environmental Protection Agency, SOP 2012 "Soil Sampling", Revision 0.0, February 18, 2000

5. Attachments

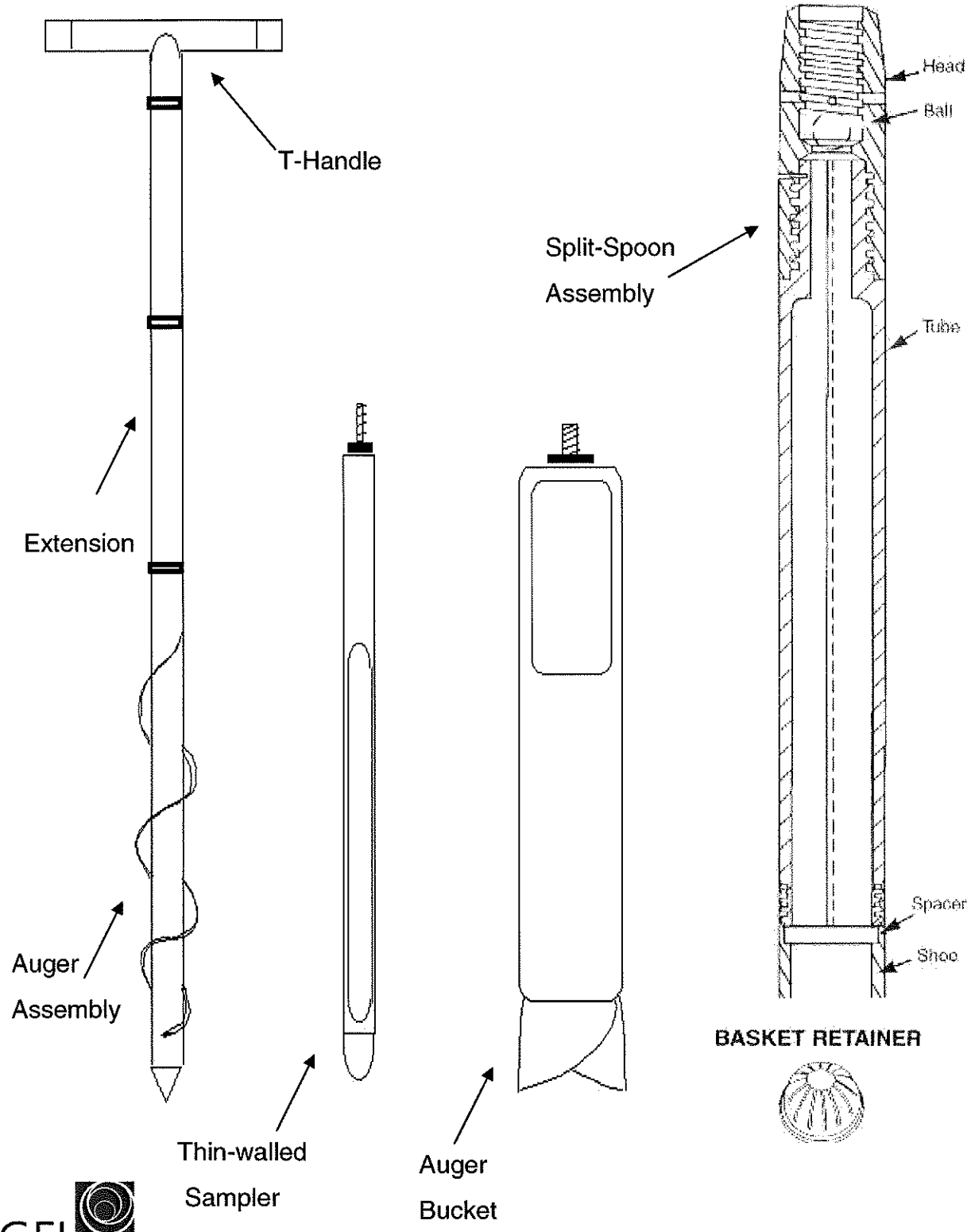
Attachment A - Sampler Design Assembly

6. Contact

Gary Fuerstenberg

SOP SM-001

Attachment A – Sampler Design Assembly



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STANDARD OPERATING PROCEDURE

SM-003 – Soil Classification

1. Objective

To describe and classify soil samples collected in the field in a consistent and useful manner.

2. Execution

- Describe soil samples according to the ASTM Standard Practice for Description and Identification of Soils (Visual-Manual Procedure) D2488 (see attached charts).
- Identify and record the soil in terms of the major and minor constituents (i.e., sand gravel, silt, clay), Unified Soil Classification Symbol, sample structure, plasticity and dilantancy for fine-grained soils, color, local or geologic name if known (e.g., Boston Blue Clay or glacial till), odor, presence of iron or other staining, and presence of organic matter, shells debris, or other unusual characteristics of the same.
- If a soil split-spoon sample contains more than one soil type (for example, the upper portion is silty sand and the lower portion is clay) describe each type separately, and obtain separate jars of each type.
- Record sampler type, blow counts, soil description, etc. on the boring log (see attachement 2 boring log).
- One modification to the ASTM standard: Use "widely graded" and "narrowly graded" instead of "well-graded" and "poorly graded."

3. Limitations

- Certain projects or clients will require the use of other classification systems. Other classification systems should not be used unless specifically required by the client.

- Some soil characteristics, such as plasticity and dilatancy, are difficult to identify in the field during extremely cold or wet weather. The field classification should be verified in the office after the samples have returned to room temperature if samples were collected during extreme weather conditions.

- The ASTM Standard Test Method for Classification Soils for Engineering Purposes, D2487 may be used in conjunction with the Visual-Manual Method to confirm the soil classification.

4. References

Annual Book of ASTM Standards (1993), Section 4, v. 4.08 Soil and Rock; Building Stones; Geosynthetics, D2488-90, Standard Practice for Description and Identification of Soils (Visual-Manual Procedure), American Society of Testing Materials (ASTM).

5. Attachments

Attachment 1 – Visual Manual Descriptions
Attachment 2 – Sample Boring Log

6. Contact

Lynn Willey

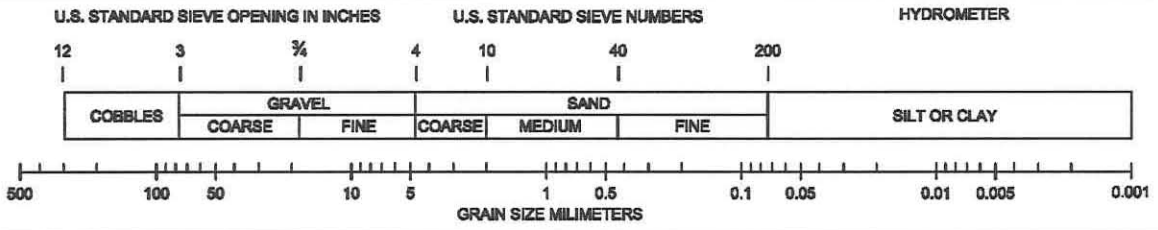
COARSE-GRAINED SOILS
VISUAL-MANUAL DESCRIPTIONS

GEI Consultants

TYPICAL SOIL COLORS

- YELLOW
- OLIVE
- LIGHT BROWN
- BROWN
- REDDISH BROWN
- RED
- GRAY
- BLACK

GROUP SYMBOL	GROUP NAME
GRAVEL (% Gravel > % Sand)	
GW	WIDELY GRADED GRAVEL
GW	WIDELY GRADED GRAVEL WITH SAND
GP	NARROWLY GRADED GRAVEL
GP	NARROWLY GRADED GRAVEL WITH SAND
GW-GM	WIDELY GRADED GRAVEL WITH SILT
GW-GM	WIDELY GRADED GRAVEL WITH SILT AND SAND
GW-GC	WIDELY GRADED GRAVEL WITH CLAY
GW-GC	WIDELY GRADED GRAVEL WITH CLAY AND SAND
GP-GM	NARROWLY GRADED GRAVEL WITH SILT
GP-GM	NARROWLY GRADED GRAVEL WITH SILT AND SAND
GP-GC	NARROWLY GRADED GRAVEL WITH CLAY
GP-GC	NARROWLY GRADED GRAVEL WITH CLAY AND SAND
GM	SILTY GRAVEL
GM	SILTY GRAVEL WITH SAND
GC	CLAYEY GRAVEL
GC	CLAYEY GRAVEL WITH SAND
SOILS WITH <50% FINES	
SAND (% Sand ≥ % Gravel)	
SW	WIDELY GRADED SAND
SW	WIDELY GRADED SAND WITH GRAVEL
SP	NARROWLY GRADED SAND
SP	NARROWLY GRADED SAND WITH GRAVEL
SW-SM	WIDELY GRADED SAND WITH SILT
SW-SM	WIDELY GRADED SAND WITH SILT AND GRAVEL
SW-SC	WIDELY GRADED SAND WITH CLAY
SW-SC	WIDELY GRADED SAND WITH CLAY AND GRAVEL
SP-SM	NARROWLY GRADED SAND WITH SILT
SP-SM	NARROWLY GRADED SAND WITH SILT AND GRAVEL
SP-SC	NARROWLY GRADED SAND WITH CLAY
SP-SC	NARROWLY GRADED SAND WITH CLAY AND GRAVEL
SM	SILTY SAND
SM	SILTY SAND WITH GRAVEL
SC	CLAYEY SAND
SC	CLAYEY SAND WITH GRAVEL



1. GROUP NAME and (SYMBOL)
2. Structure , if any. (stratified layer thicknesses, lenses, varves, gradational changes)
3. Describe sand, gravel and fines components, with percentages, in order of predominance. Include max gravel size. For test pits give percent cobbles and boulders, by volume, and include max size.
4. Color
5. Sheen, odor, roots, ash, brick, cementation, reaction with HCL, etc.
6. "Fill," local name or geologic name, if known

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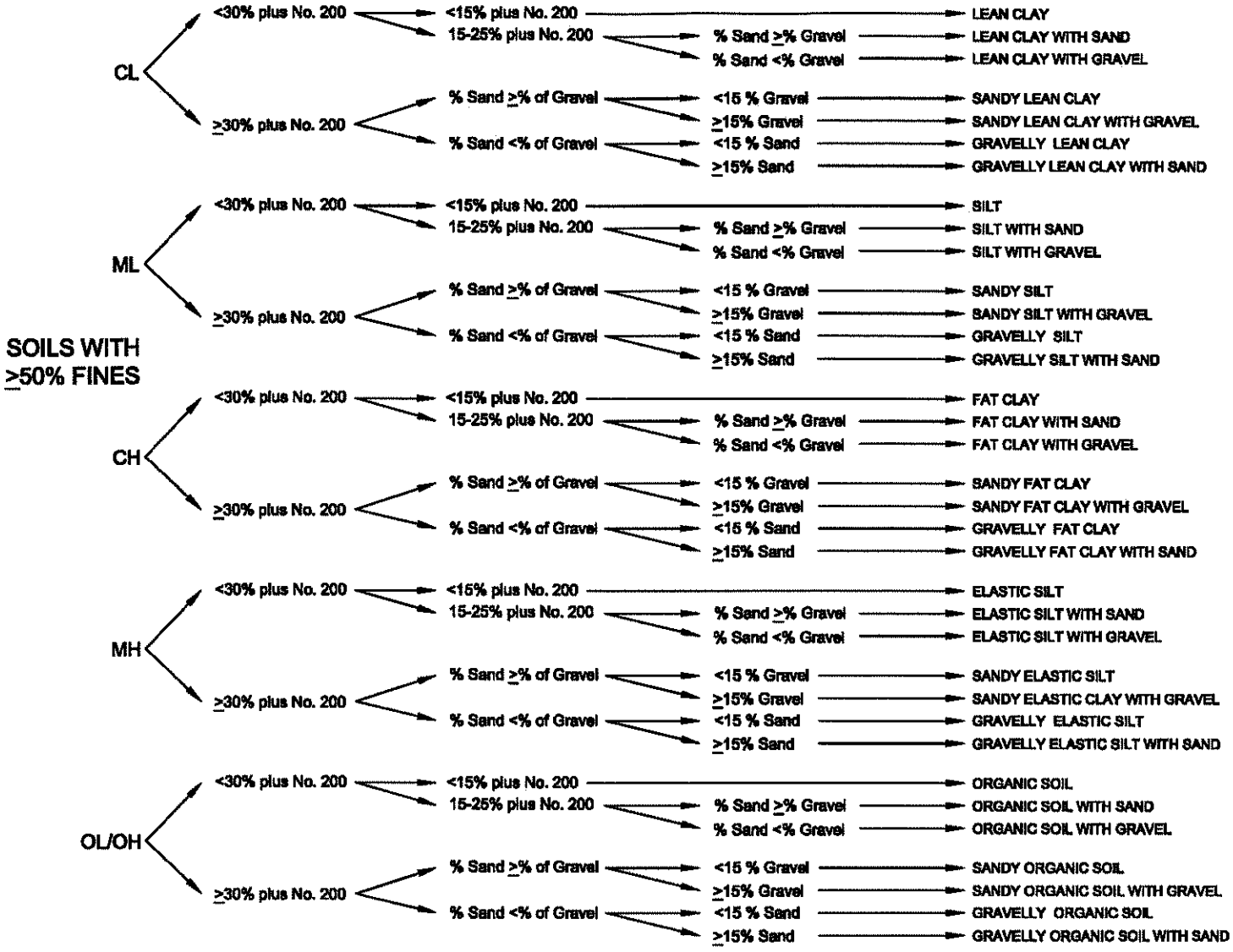
FINE-GRAINED SOILS
VISUAL-MANUAL DESCRIPTIONS



ID OF INORGANIC FINE SOILS FROM MANUAL TESTS

Symbol	Name	Dry Strength	Dilatancy	Toughness*
ML	Silt	None to low	Slow to rapid	Low or thread cannot be formed
CL	Lean Clay	Medium to high	None to slow	Medium
MH	Elastic Silt	Low to medium	None to slow	Low to medium
CH	Fat Clay	High to very high	None	High

1. GROUP NAME and (SYMBOL)
2. Describe fines, sand, and gravel components, in order of predominance. Include plasticity of fines. Include percentages of sand and gravel.
3. Color
4. Sheen, odor, roots, ash, brick, cementation, torvane and penetrometer results, etc.
5. "Fill," local name or geologic name, if known

PEAT

Peat refers to a sample composed primarily of vegetable matter in varying stages of decomposition. The description should begin: PEAT (PT) and need not include percentages of sand, gravel or fines.

CRITERIA FOR DESCRIBING PLASTICITY

Description	Criteria
Nonplastic ML	A 1/8-in. (3 -mm) thread cannot be rolled at any water content
Low Plasticity ML, MH	The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit *
Medium Plasticity MH, CL	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit
High Plasticity CH	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rerolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit

* Toughness refers to the strength of the thread near plastic limit. The lump refers to a lump of soil drier than the plastic, similar to dry strength.

BORING LOCATION Maple Ave Sidewalk
GROUND ELEVATION (NGVD) _____
GROUNDWATER EL. _____ DATE _____
DATE START/FINISH 2/14/07 - 2/15/07
DRILLED BY Geologic: M. Costigan
LOGGED BY T. Kahl/M. Yako TOTAL DEPTH (FT) 25

B101

PG. 1 OF 1

EL. FT.	DEPTH FT.	SAMPLE TYPE and NO.	SAMPLE BLOWS PER 6 IN.	SAMPLE PEN IN.	SAMPLE REC IN.	PID JAR HS / REMARKS	GRAPHIC LOG	SOIL AND ROCK DESCRIPTIONS
	0						FILL	4" pavement
		S1	13-9 17-14	24	0	0.5 ppm		S1: Redrove 0.5 to 3.5 ft. Recovery 11": WIDELY GRADED SAND (SW) ~85% sand, ~10% gravel to 1", <5% nonplastic fines, brown. Contains brick fragments and ash. Fill.
	2.5					hard drilling 3 to 4 ft, possible boulder		
	5	S2	7-7 11-13	24	8	2.0 ppm		S2: NARROWLY GRADED SAND WITH SILT AND GRAVEL (SP-SM) ~65% mostly fine sand, ~25% gravel to 3/4 inch ~10% non-plastic fines, brown. Fill.
	7.5	S3	9-10 2-1	24	16	0.0 ppm		S3 (0-10"): Similar to S2.
	10						ORGANICS	S3 (10"-16"): ORGANIC SILT (OL) ~100% slightly plastic fines, dark gray, organic odor, contains white shell fragments.
	12.5	S4	WOH 1-2 1	24	15	0.0 ppm		
	15					hard drilling at 15.5 ft		S4: Similar to S3, bot 6".
	17.5	S5	20-35 50/3"	15	8	Top of rock ~19 ft. Roller bit to 20 ft.	TILL	S5: SILTY SAND WITH GRAVEL (SM) ~60% mostly fine sand, ~25% slightly plastic fines, ~15% gravel to 1/2 inch, olive. Glacial Till.
	20							
	22.5	C1	RQD 70%	60	54	lost ~10 gallons drill fluid from 23 to 25 ft	ROCK	C1: SCHIST, hard, slight weathering at joint surfaces, joints at ~30 degrees from horizontal and generally parallel to foliation, gray. Marlborough Formation.
	25							Bottom of Boring 25 ft
	27.5							Truck-mounted drill rig. 4-inch casing to 19 ft. Safety-hammer with rope and cathead for SPT. Backfilled with drill cuttings.
	30							

BLOWS PER 6 IN.–140 LB. HAMMER FALLING 30 IN. TO DRIVE A 2.0 IN. OD SPLIT SPOON SAMPLER
PEN–PENETRATION LENGTH OF SAMPLER OR CORE BARREL
REC–RECOVERY LENGTH OF SAMPLE
RQD–LENGTH OF SOUND CORES > 4 IN./ LENGTH CORED, %
S–SPLIT SPOON SAMPLE
U–UNDISTURBED SAMPLES, UF–FIXED PISTON UO–OSTERBERG
▽ GROUNDWATER

NOTES:
1: Groundwater at 10 ft depth at start of day 2/15/07.

PROJECT 07999-0
DATE

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EXAMPLE SOIL DESCRIPTIONS

SANDY SILT (ML) ~60% slightly plastic fines, ~40% mostly fine sand, 1" thick layer of fine to medium sand with <20% fines, gray.

LEAN CLAY (CL) ~90% moderately plastic fines, ~10% fine sand, olive. Boston Blue Clay. Sv = 0.5, 0.5, 0.8 tsf, Qp = 1.0, 1.5, 1.6 tsf

Stratified CLAYEY SAND (SC) and WIDELY GRADED SAND (SW) SC layers 1 to 2 inches thick consist of fine sand with ~30% moderately plastic fines, gray. SW layers 1 to 4 inches thick consist of fine to coarse sand, ~10% gravel to 1/2 inch, <5% fines, brown. Hydraulic Fill.

EXAMPLE ROCK DESCRIPTIONS

(0-9"): GRANITE, hard, one piece, joint surface slightly weathered, pink.
(6-60"): PHYLLITE, joints ~ 45° generally parallel to foliation, 9" to 44" moderate to severe jointing and joint weathering. 44" to 60" single piece, green-gray.

ARGILLITE, medium hard, moderately weathered joints, gray. Cambridge Argillite.

GEOPROBE AND ROTOSONIC

When SPTs are not performed, note sample density (sands) or stiffness (clays) in description.

CRITERIA FOR DESCRIBING DILATANCY OF FINE-GRAINED SOILS

Description	Criteria
None	No visible change in the specimen
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing.
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing.

SPT: Standard Penetration Test

30-inch drop with 140-lb hammer
1 3/4 to 2 1/4 turns around cathead
2-inch O.D. split spoon sampler

ENV'L TERMINOLOGY FOR SOIL DESCRIPTIONS

- **Ash** - Typically silt-size to medium sand-size.
- Do not use the term "cinders." This is not a technical term. Instead, use "ash," "burnt wood," "burnt material," or a similar term.
- **Coal-like material** - If it looks like coal but you aren't sure.
- **Clinker** - Vitrified (glass-like) or heat-fused material. Often burned impurities in coal. Often looks like pumice, but heavier.
- **Slag** - Similar to clinker, but normally refers to residue from metal ore processing.

- **Sheen** - Iridescent petroleum-like sheen. Not to be used for a "bacterial sheen," which can be distinguished by its tendency to break up on the water surface at angles. Petroleum sheen will be continuous and will not break up.
- **Stained** - Use with a color ("brown-stained") to indicate that the soil is stained a color other than its natural (unimpacted) color.
- **Coated** - Soil grains are coated with NAPL (oil, tar, etc.). There is not enough NAPL to saturate the pore spaces. ("Split spoon sampler coated with brown oil." "Soil grains coated with gray substance with slight gasoline-like odor.")
- **Saturated** - The entire sample pore space is saturated with NAPL. If you use this term, be sure it is not water saturating the pore spaces. Depending on viscosity, the NAPL may drain from a soil sample. ("Sample saturated with green, sticky substance.")
- **Blebs** - Discrete sphericals of NAPL in a soil matrix that was not visibly coated or saturated. ("Occasional blebs of reddish-brown tar.")
- **Oil** - Exhibits a petroleum odor, different from MGP odors.
- **Tar** - Exhibits an MGP odor (e.g. naphthalene-like odor).

- **Odors** - Use terms such as "naphthalene-like odor" or "petroleum-like odor." Use modifiers (strong, moderate, slight) to indicate odor intensity.

STANDARD OPERATING PROCEDURE

SM-007 – Chip Sampling

1. Objective

The objective of this SOP is to standardize the collection and sampling of concrete samples for environmental investigations.

2. Execution

- Contact the Owner to determine the locations of underground utilities/obstructions.
- For easy identification, sample locations may be pre-marked using a crayon or a non-contaminating spray paint. (Note, the actual drilling point must not be marked.) Depending on the appearance of the sample location, or the objectives of the sampling project, it may be desired to wipe the concrete surface with a clean dry cloth prior to drilling. All sampling decisions of this nature should be noted in the sampling logbook (See Precautions Below).

2.1. Concrete Dust Sampling (Not appropriate for VOC sampling)

- Lock a 1-inch diameter carbide drill bit into the impact hammer drill and plug the drill into an appropriate power source. (A gasoline generator will be needed if electricity is not available.)
- Begin drilling in the designated location. Apply steady even pressure and let the drill do the work. Applying too much pressure will generate excessive heat and dull the drill bit prematurely. The drill will provide a finely ground concrete powder that can be easily collected, homogenized and analyzed. Having several decontaminated impact drill bits on hand will help expedite sampling when numerous sample locations are to be drilled.
- A 2-inch deep hole (using a 1-inch diameter drill bit) generates about 10 grams of concrete powder. Based on this and the action levels for the project, determine the sampling depth, and/or the number of sample holes to be composited, to generate sufficient sample volume for all of the required analyses. (Note, with the absorbency of concrete, a 2-inch deep hole can be considered a surface sample.
- A decontaminated stainless steel scoop can be used to collect the sample. The powder can either be collected directly from the surface of the concrete and/or the concrete powder can be

scraped back into the hole and the less rounded back edge of the scoop can be used to collect the sample. For holes greater than 2-inches in depth, a stainless steel spoon will make it easier to collect the sample from the bottom of the hole.

- To ensure collection of a representative sample when multiple analyses are required, a concrete sample should always be collected and homogenized in a single container and then divided up into the individual containers for the various analyses or split samples. This is particularly important when sample holes are deep, or when several holes are drilled adjacent to each other to form a sample composite.

2.2. Concrete Chip Sampling

- If possible, remove any non-porous inclusions from the sampling location by brushing or wiping, as appropriate.
- Using a chisel, drill, hole saw, or similar tool, collect a minimum of 100 g of the sample to a depth of 2 cm, or to an alternate depth specified in applicable planning documents. The collected chips may be of any convenient size unless otherwise specified in applicable planning documents.
- Transfer the sample to an appropriate sample container. SC-002 Sample Handling, provides guidance regarding the amount of sample, the type of sample container, the holding time, and the preservation techniques to be used for each analysis to be conducted. Complete Sample Collection Logs and Chain of Custody Forms; label sample containers and complete documentation.

3. Limitations

- Concrete sampling may require removing tiles or laminent coverings with asbestos containing adhesives. These coverings should not be removed without a determination of the presence of asbestos. If asbestos is present, the material will need to be removed prior to any concrete sampling.
- If collecting multiple samples using this method, avoid cross-contamination by decontaminating all sampling tools prior to collecting the next sample. If the sampler's gloves come in contact with the sampled material during sampling, gloves should also be changed prior to collecting the next sample.

4. References

Draft Standard Operating Procedure for Sampling Concrete in the Field, Environmental Protection Agency, Region 1, December 1997.

Environmental Restoration Project Standard Operating Procedure for Los Alamos National Laboratory, Los Alamos National Laboratory, December 2001.

5. Attachments

None

6. Contact

Lynn Willey

STANDARD OPERATING PROCEDURE

GW-001 Water Level Measurement

1. Objective

To obtain accurate repeatable water level readings for determination of groundwater flow direction.

2. Execution

- Prior to collecting water level measurements all wells should be opened to the atmosphere and allowed to equilibrate prior to collecting groundwater elevation measurements.
- All groundwater level measurements need to be performed in the shortest possible timeframe (no more than four hours).
- Groundwater levels are measured using a electronic ground water-level indicator which have a cable divided into incremental measurements of 0.01 feet and two conductors forming a probe. When ground water is encountered, the circuit is completed and a light, meter or audible buzzer is activated. The depth to ground water is then measured from this point to the reference mark on the inner casing of the monitor well.
- All ground water-level measurements should be made from the same marked reference point at the top of the inner well casing. A licensed surveyor must mark the reference point.
- If no discernable survey mark is observed on the inner casing, the ground water-level measurement should be read from the highest point of the inner casing.
- If no survey mark is observed on the inner casing, it should be noted with the ground water-level data and the highest point of the casing must be marked for future reference.
- Measurements should be made three to four times to confirm the measurement. Each time a measurement is made it should be determined to the nearest one-hundredth of a foot (0.01).
- All well measurements should be performed the same day, and prior to the evacuation of any wells which may influence groundwater elevations in the area of the investigation.
- Measurements should be collected from the same survey point, and to avoid any procedural differences, preferably by the same person and measuring tape.
- The following items should be recorded on field data sheets while collecting groundwater level measurements:
    1. Diameter of protective outer casing
    2. Security and integrity of the well
    3. Well number

4. Inner diameter and construction material of the inner well casing
5. Total depth of the well from the top of the inner casing or surveyor's mark, if present (measured to 0.01 foot)
6. Depth from the top of the inner casing to ground water (recorded to 0.01 foot accuracy)
7. Thickness of floating product, if any. See SOP GW-002 NAPL Measurement
8. Calculation of the linear feet of water in the well by subtracting the depth to ground water from the total depth of the well.
9. Calculation of the water table elevation in the well by subtracting the depth to ground water from the top-of - casing elevation.

3. Limitations

- Groundwater levels should be obtained from all wells in a network prior to sampling the first well.
- All wells should be sampled .
- Weak batteries in these units frequently produce weak or gradual auditory and/or visual responses, making it difficult to accurately determine when the probe of the unit has come in contact with ground water. As such, it is recommended that electronic ground water-level indicators be tested before they are brought out into the field.
- Note that electronic ground water-level indicators will not respond to distilled water, so distilled water should not be used to test these units.
- Wells that are not plumb may result in probe contact with the side of the well casing providing a false measurement. Once the probe has come in contact with ground water in the well, water may be trapped by capillary action between the probe and the well casing. If this happens, the unit may continue to signal even after the probe has been raised above the ground water surface. The deeper the well, the more likely this problem may occur. To correct this, the cable should be raised several feet above the water and shaken to remove water from the probe. A new ground water-level measurement should then be collected. If the signals from the unit are not abrupt or reproducible, the probe may need to be reeled up to the surface and dried off before re-attempting another measurement.
- Accumulation of sediment, organic material, or floating debris on the probe may also result in gradual or non-reproducible readings. Wells that are constructed with metal inner casings may lead to

difficulties in collecting reproducible ground water-level measurements because the inner sides of the well casing are conductive.

- In some cases, a rubber grommet or metal centralizer may need to be placed on the probe so that the probe is not allowed to come in contact with the inner casing. Ground water-level-measuring equipment should be properly decontaminated between wells and piezometers to avoid cross contamination.
- Once a well has been located and properly identified, the field measurements listed below should be noted in a field logbook. Be certain that the proper well is being measured. The misidentification of a sampling point in the field will result in erroneous data that may result in incorrectly constructed contour maps.

4. References

None

5. Attachments

Attachment A – Monitoring Well Sampling Record

6. Contact

Brian Conte

GEI Consultants

MONITORING WELL SAMPLING RECORD

PID Reading _____ Job Name _____

Job Number _____ By _____ Date _____

Location _____ Measurement Datum _____

Well Number _____

Pre-Development Information Time (start) _____

Water Level _____ Total Depth of Well _____

One Purge Vol _____ Three Well Volume _____

Water Characteristics

Color _____ ___ Clear ___ Cloudy

Odor ___ None ___ Weak ___ Moderate ___ Strong

Any films or immiscible material ___ None

Volume (gal)	Time	pH	Temp (EC)	Spec. Conductance (ΦS/cm)	Turbidity (NTU)	DO Conc. (mg/L)	ORP (mV)	TDS

Total Volume Removed (gal) _____ pH _____

Temperature (EC) _____ Specific Conductance (ΦS/cm) _____

DO Concentration (mg/L) _____ ORP (mV) _____

_____ TDS _____

Post Development Information Time (Finished) _____

Water Level _____ Total Depth of Well _____

Approximate Volume Removed (gal) _____

Water Characteristics

Color _____ ___ Clear ___ Cloudy

Odor ___ None ___ Weak ___ Moderate ___ Strong

Any films or immiscible material ___ None

_____

Comments:

STANDARD OPERATING PROCEDURE

GW-002 Light Non-Aqueous Phase Liquid (LNAPL) Measurement

1. Objective

Accurate and repeatable measurement of the thickness of light non-aqueous phase liquids (LNAPL) contained in monitoring wells.

2. Execution

Two procedures for measuring LNAPL are provided below: clear bailer and Interphase probe. Neither method is ideal, however, due to difficulties associated with the Interface Probe, use of the clear bailer is the preferred method to identify and estimate thickness of floating product in monitor wells.

2.1. Clear Bailer

- Determine depth to the surface level of the LNAPL layer.
- Record depth.
- Lower a clear bailer into the well and slowly into the product, being careful not to submerge the bailer.
- Raise the bailer and measure product thickness.
- Once the product thickness is known, the depth to ground water may be determined (See calculation below).
- This method has inaccuracies because successful use of the bailer is dependent upon the expertise of the operator and assumes the check valve does not leak upon retrieval.

2.2. Interface Probes

- Decontaminate interphase probe prior to use.
- Check battery and replace if necessary.
- Check the unit is functioning correctly. Note: De-ionized water will not provide a correct reading.
- Measure the hydrocarbon/air interface first by going from air to the LNAPL surface to prevent dripping hydrocarbons from enhancing the thickness reading.
- Record reading
- Measure the hydrocarbon/water reading by lowering the interphase probe past the LNAPL layer quickly minimizing the contact time of the probe within the hydrocarbon phase.
- DNAPL can also be measured by quickly lowering the interphase probe past the LNAPL layer and to the bottom of the well noting any audio or visual indications of DNAPL.
- Optical sensor on interface probes may become damaged if solvents are used to clean product from the probes.

- Optical sensor may become smeared when used to measure product, rendering pinpoint accuracy to an estimate at best.
- Close attention to decontamination procedures will improve accuracy, operational life and reduce the risk of cross contamination with other wells.

3. Limitations

- When an LNAPL thickness is measured in a monitoring well it will usually exhibit an apparent thickness rather than an actual thickness. This apparent thickness is caused when LNAPL from within and above the capillary fringe migrates into the monitoring well causing the ground water-level to become depressed below the surrounding capillary fringe area. As a result, LNAPL will continue to flow into the well until equilibrium is reached causing an apparent LNAPL thickness, which is greater than the actual thickness.
- LNAPL thickness can be affected by fluctuations in the water table. In some cases, an LNAPL's thickness may decrease when the water table rises, while its thickness increases as the water table drops. In other cases, fluctuating water tables may cause sudden appearances and disappearances of LNAPL layers.
- Monitoring points with Light Non-aqueous Phase Liquids (LNAPLs) can pose a problem when measuring the level of ground water. Floating LNAPLs can depress the ground water-level in a monitoring well or piezometer and distort the measurement. Therefore, the corrected depth (CD) formula shown below should be applied to ground water-level measurements in monitoring points where LNAPLs are present:

CDTW = Static DTW – (PT x G)

CDTW = Corrected Depth to Ground water
DTW = Depth to Ground Water (Static)
PT = Measured Product Thickness
G = Specific Gravity (density of free product / density of water)

4. References

None

5. Attachments

Sample Groundwater Monitoring Sheet

6. Contact

Brian Conte

GEI Consultants

MONITORING WELL SAMPLING RECORD

PID Reading _____ Job Name _____

Job Number _____ By _____ Date _____

Location _____ Measurement Datum _____

Well Number _____

Pre-Development Information Time (start) _____

Water Level _____ Total Depth of Well _____

One Purge Vol _____ Three Well Volume _____

Water Characteristics

Color _____ ___ Clear ___ Cloudy

Odor ___ None ___ Weak ___ Moderate ___ Strong

Any films or immiscible material ___ None

Volume (gal)	Time	pH	Temp (EC)	Spec. Conductance (ΦS/cm)	Turbidity (NTU)	DO Conc. (mg/L)	ORP (mV)	TDS

Total Volume Removed (gal) _____ pH _____

Temperature (EC) _____ Specific Conductance (ΦS/cm) _____

DO Concentration (mg/L) _____ ORP (mV) _____

_____ TDS _____

Post Development Information Time (Finished) _____

Water Level _____ Total Depth of Well _____

Approximate Volume Removed (gal) _____

Water Characteristics

Color _____ ___ Clear ___ Cloudy

Odor ___ None ___ Weak ___ Moderate ___ Strong

Any films or immiscible material ___ None

_____

Comments:

STANDARD OPERATING PROCEDURE

GW-003 Low Flow (Low Stress) Groundwater Sampling

1. Objective

Outline a method to collect groundwater samples that accurately and precisely represent the aquifer conditions. Low-flow purging is limited to wells that, with sustained pumping, exhibit no continuous drawdown.

2. Execution

- Record all activities in the field notebook (see SOP FD-001 Field Notebook SOP) and on Attachment A - Low-Flow Groundwater Sampling Form (attached). Use a separate form for each sampling location and event.
- Calibrate PID, pH, temperature, specific conductance (SC), turbidity, dissolved oxygen (DO), and oxidation-reduction potential (ORP) meters.
- Start at the well known or believed to have the least contaminated groundwater and proceed systematically to the well with the most contaminated groundwater. Check the well, the lock, and the locking cap for damage or evidence of tampering.
- Record observations.
- Being careful to not disturb the water column, slowly and gently measure the depth to water with a water level probe and/or oil water interface probe. Do not measure depth to well bottom at this time (wait until sampling has been completed). Measure water level to the nearest 0.01 foot from the top of casing and the highest point (or "V" notch) on the PVC. If the top of casing cannot be used, note the reference location. Mark the datum point with an indelible marker and note reference location in field book.
- Attach in-line flow-through cell at discharge end of pump and provide shade for flow-through cell.
- Slowly and gently insert the pump intake tubing to the middle of the saturated screened interval, open borehole or to the pre-determined sampling depth. The pump intake must be kept at least two (2) feet above the bottom of the well to prevent disturbance or suspension of any sediment or NAPL present in the bottom of the well. Record the depth of the pump intake.
- Start the pump on the lowest setting and increase slowly until flow begins. Adjust the pumping rate so that drawdown in the well is minimal (0.3 feet or less). Use a pumping rate between 100 to 1,000 milliliters per minute (mL/min) [or approximately 0.1 to 1 quarts per minute]. Measure rates with a graduated container

every 3 to 5 minutes and record. The minimum purge volume will be twice the combined volume of the sampling string (pump, tubing, flow-through cell).

- While purging, record water levels every 3 to 5 minutes. A steady state flow rate will be maintained that results in a stabilized water level with a drawdown of 0.3 feet or less.
- After pumping at least two volumes of the sampling string, monitor and record every 3 to 5 minutes the water quality indicator parameters that include: pH, temperature, specific conductance, and turbidity. If specified in the field sampling plan, also include DO and ORP.
- Purging is complete when, after three consecutive measurements, the water quality parameters have stabilized as follows:
    1. pH (+/- 0.2 S.U.)
    2. temperature (+/- 0.2 C)
    3. SC (+/- 5% umhos/cm)
    4. turbidity (+/- 5 NTU)
    5. DO (+/- 0.2 mg/L or 10%, whichever is greater)
    6. ORP (+/- 20 mV or 10%, whichever is greater)
- Dispose of purge water according to the field plan.
- Collect Samples
- Following purge, disconnect the flow-through cell and fill all containers from the discharge end of the tubing. Collect samples at a flow rate equal to or less than the steady state purge rate.
- Fill sample containers directly from the sampling device in order of decreasing volatility (i.e., VOC samples will be collected first; see SOP SC-002 Sampling Handling).
- If not using dedicated equipment, remove sampling device and decontaminate (see SOP QA-001 Equipment Decontamination).
- Store samples in cooler between 2°C and 6°C for transport to the laboratory.
- Measure depth to bottom of well.
- Secure the well cap.

3. Limitations

- At all times, follow safety procedures as defined in the site-specific Health and Safety Plan.
- Prior to departure for the field, obtain available information on well construction for use in field investigation (i.e., screen and riser material, well diameter and depth, screened interval, optimum sampling depth, etc.).

- If using dedicated equipment, install equipment into well at least 24 hours before sample collection to minimize disturbance of the water column and/or suspension of sediments or NAPL on bottom.
- Measure the depth to water twice.
- Measure the well bottom after sampling to avoid disturbance of water column and/or suspension of sediments or NAPL at the bottom of the well.
- To prevent cross-contamination between wells, it is strongly recommended that dedicated, in-place pumps (and tubing) be used.
- If the water quality indicator parameters do not stabilize after 2 hours, then either continue purging or, contact the Project Manager.
- The key indicator parameter for VOCs is DO. The key indicator parameter for all other samples is turbidity.
- Turbidity and DO usually require the longest time to achieve stabilization. The pump must not be removed from the well between purging and sampling.
- All sample containers are to be filled with minimal turbulence by allowing the groundwater to flow from the tubing gently down the inside of the container.
- Field filtering for metals is not necessary if using low-flow sampling (turbidity must be less than 5 NTU). Field filtering is not allowed unless authorized under the project sampling plan.
- Be aware of any preservatives in the sample bottles and handle with care, in accordance with the Health and Safety Plan.

4. References

Standard Reference for Monitoring Wells (April 19, 1991), Massachusetts DEP, DEP Publication No. WSC-310-91.

Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures (1996), R.W. Puls and M.J. Barcelona, U.S. Environmental Protection Agency, EPA/540/S-95/504.

Reproducible Well-Purging Procedures and VOC Stabilization Criteria for Ground Water Sampling (1994), M.J. Barcelona, H. A. Wehram, and M.D. Varljen, Ground Water, Vol. 32, No. 1, 12-22.

Low-Flow Purging and Sampling of Ground Water Monitoring Wells with Dedicated Systems (1995), R.W. Puls, and C.J. Paul, Groundwater Monitoring and Review, Summer 1995 116-123.

Ground Water Sampling Procedure Low Stress (Low Flow) Purging and Sampling, (1998), Ground-Water Sampling SOP, Final, U.S. Environmental Protection Agency, Region II, March 16, 1998.

RCRA Ground-Water Monitoring: Draft Technical Guidance, (1993), U.S. Environmental Protection Agency, EPA/530-R-93-001.

To Filter, or Not to Filter, That is the Question, (1997), Special Topics Subcommittee Letter Report EPA-SAF-EEC-LTR-97-011, April 29, 1997, Meeting, U.S. Environmental Protection Agency, Science Advisory Board Environmental Engineering Committee, September 5, 1997.

Should Filtered or Unfiltered Groundwater and Surface Water Samples be Collected for the Risk Assessment?, (1995), MCP Q&A: Subparts I and J, Special #4, Bureau of Waste Site Cleanup, Massachusetts Department of Environmental Protection (DEP), February, 1995.

5. Attachments

Attachment A - Sample Groundwater Monitoring Sheet

6. Contact

Brian Conte

GEI Consultants

MONITORING WELL SAMPLING RECORD

PID Reading _____ Job Name _____

Job Number _____ By _____ Date _____

Location _____ Measurement Datum _____

Well Number _____

Pre-Development Information Time (start) _____

Water Level _____ Total Depth of Well _____

One Purge Vol _____ Three Well Volume _____

Water Characteristics

Color _____ Clear ____ Cloudy ____

Odor ____ None ____ Weak ____ Moderate ____ Strong

Any films or immiscible material ____ None

Volume (gal)	Time	pH	Temp (EC)	Spec. Conductance (ΦS/cm)	Turbidity (NTU)	DO Conc. (mg/L)	ORP (mV)	TDS

Total Volume Removed (gal) _____ pH _____

Temperature (EC) _____ Specific Conductance (ΦS/cm) _____

DO Concentration (mg/L) _____ ORP (mV) _____

_____ TDS _____

Post Development Information Time (Finished) _____

Water Level _____ Total Depth of Well _____

Approximate Volume Removed (gal) _____

Water Characteristics

Color _____ Clear ____ Cloudy ____

Odor ____ None ____ Weak ____ Moderate ____ Strong

Any films or immiscible material ____ None

_____

Comments:

STANDARD OPERATING PROCEDURE

GW-004 pH and Temperature Measurement

1. Objective

The objective of this Standard Operating Procedure is to provide standard methods for determining the pH and temperature of liquids using a combination pH/temperature meter.

2. Execution

- Calibrate the meter according to the equipment manufacturer's instructions at the beginning of each day of use. Calibration for pH shall be performed using at least two buffer solutions from various ranges. Solutions chosen should be similar in pH to the expected level of the samples or liquids tested (pH 7 and 4 buffer solution preferred in most cases for ground or surface water measurements).
- Calibration is checked every two hours or every five monitoring locations (whichever occurs first) and at the end of the day be measuring the two calibration solutions used. The reading and times are recorded. If the readings are outside ∀0.2 pH units, the meter must be recalibrated.
- Immediately prior to testing a sample decontaminate testing beaker or container and probe assembly with one rinse with sample solution. Do not use methanol to rinse the probe. Methanol rinses could damage the probe.
- Gently shake the probe and beaker to remove excess solution. Visually inspect the bottom of the probe to ensure that liquid or sediment is not trapped between outer casing and probe.
- Pour sample into testing container and insert both temperature and pH probe. Stir sample for 30 seconds using both probes. Let the probes equilibrate in the sample solution for another 30 seconds. Measure and record the temperature. Measure and record pH reading after the reading has stabilized or after 60 seconds, whichever is sooner. A reading has stabilized if pH units have not changed ∀0.1 pH units during a five second period.
- Record pH to the nearest 0.1 unit and temperature to the nearest whole number.

3. Limitations

- At all times, follow safety procedures as defined in the site-specific Health and Safety Plan.

- Only coatings and particulates may affect the response of the probe; more thorough cleaning with alcono and distilled water and gently wiping the probe surface may be required to clean the surface of the probe.
- Temperature affects both the response of the instrument to pH and the actual pH of the sample. The automatic temperature compensation (ATC) function compensates for the variation in the response of the meter only. Therefore, the pH must always be reported with temperature.
- The probe is a fragile thin glass bulb surrounded on three sides by a plastic casing. Care must be taken in handling the probe to avoid breakage.
- Buffer solutions should not be used past their expiration date.

4. References

Standard Methods for the Examination of Water and Wastewater, 18th Edition, Method 4500-H. American Public Health Association (1992).

5. Attachments

None

6. Contact

Brian Conte

STANDARD OPERATING PROCEDURE

GW-005 Turbidity Measurement

1. Objective

A nephelometer/ turbidimeter is used in comparing the turbidity of liquids by viewing light through them and determining how much light is eliminated. Turbity readings are required to be read using a portable (e.g Hach) instrument outside the flow through cell.

2. Execution

- Turn the meter "ON".
- Rinse the sample cell 3 times with organic free or de-ionized water.
- Fill the cell to the fill line with organic free or de-ionized water and then cap the cell.
- Use a non-abrasive lint-free paper or cloth (preferably lens paper) to wipe off excess water and streaks.
- Open the cover and insert the cell (arrow to the front) into the unit and close the cover.
- Press "READ" and wait for the 'light bulb' icon to go off. Record the reading.
- Using the Gelex standards, repeat steps above. Record all measurements (note anomalies).
- Collect a representative sample or use a portion of the sample that is collected for pH, temperature, or conductivity analysis, and pour off enough to fill the cell to the fill line (about 15 mL) and replace the cap on the cell.
- Wipe off excess water and any streaks with a soft, lint-free cloth (lens paper).
- Press I/O and the instrument will turn on. Place the meter on a flat, sturdy surface. Do not hold the instrument while making measurements.
- Insert the sample cell in the in the instrument so the diamond or orientation mark aligns with the raised orientation mark in the front of the cell compartment. Close the lid.
- Select manual or automatic range selection by pressing the range key.
- Select signal averaging mode by pressing the Signal Average key. Use signal average mode if the sample causes a noisy signal (display changes constantly).
- Press Read. The display will show ---- NTU. Then the turbidity in NTU. Record the result after the lamp symbol turns off.

- Rinse the cell with de-ionized water..
- Operational check:
- Periodically check the turbidity meter during the day by using the gelex secondary standards provided.
- Perform a post calibration at the end of the day and record all measurements.

3. Limitations

- At all times, follow safety procedures as defined in the site-specific Health and Safety Plan.
- Turbidity measurements are reported in nephelometric turbidity units (NTUs). It is important to note that if the turbidity measurements are for NPDES reporting purposes, all values above 40 NTU must be diluted with turbidity free water and calculated by multiplying by a dilution factor.

4. References

Standard Methods for the Examination of Water and Wastewater, 18th Edition, Method 4500-H. American Public Health Association (1992).

5. Attachments

None

6. Contact

Brian Conte

STANDARD OPERATING PROCEDURE

GW-006 Specific Conductance Measurement

1. Objective

The objective of this Standard Operating Procedure is to provide standard methods for determining the conductivity of waters using a field conductivity meter.

2. Execution

- Calibrate the meter according to equipment manufacturer's instructions at the beginning of each day of use. Calibration shall be performed using a standard KCL solution of 0.20 mS/cm (200 uS/cm, S=mho).
- Record the make, model, and GEI identification number of the instrument in the field notebook.
- Calibration is checked at the beginning of the day immediately prior to sampling, after five sampling locations or two hours (whichever occurs first), and at the end of the day. If the readings are outside +/- 0.02 mS/cm, the meter must be recalibrated. Initial calibration should be conducted under the same conditions (i.e., temperature, location) of field testing.
- Immediately prior to testing a sample, decontaminate testing beaker or container and probe assembly with one methanol rinse, two distilled water rinses, and one sample solution rinse.
- Gently shake the probe and beaker to remove excess solution.
- Pour sample into the testing container and insert probe. Stir sample with the probe for approximately 10 seconds. Let the probe equilibrate in the sample solution for another 5 seconds. Measure conductivity and record in field notebook.
- Record conductivity to the nearest whole number.

3. Limitations

- At all times, follow safety procedures as defined in the site-specific Health and Safety Plan.

- At all times, follow safety procedures as defined in the site-specific Health & Safety Plan.
- Oily coatings and particulates may affect the probe's response; more thorough cleaning using a weak alconox solution and double distilled water rinse and gently wiping the probe surface may be required to clean the surface of the probe.

- In contaminated example (stained, conductance >750 □mhos/cm), rinse probe with clean water immediately after measuring sample to minimize fouling of probe.
- Calibration solutions should not be used past their expiration date and must be discarded after three months of use.

4. References

Standard Methods for the Examination of Water and Wastewater, 18th Edition, Method 4500-H. American Public Health Association (1992).

5. Attachments

None

6. Contact

Brian Conte

STANDARD OPERATING PROCEDURE

GW-007 Dissolved Oxygen Measurement

1. Objective

To accurately quantify oxygen dissolved in water.

2. Execution

- It is important that the instrument be placed in the intended operating position vertical, tilted, or on its back - before it is prepared for use and calibrated.
- Readjustment may be necessary when the instrument operating position is changed.
- Attach the prepared probe to the PROBE connector of the instrument and adjust the retaining ring finger tight. (Probe is prepared during routine maintenance)
- Before calibrating, allow 15 minutes for optimum probe stabilization.
- Calibrate meter according to the procedures outline in the operation manual.
- Adjustment may be necessary for altitude greater than 1.500-feet above sea level. If so, see correction tables below for correct factor.
- Perform Dissolved Oxygen Measurement using the following procedure:
    1. Submerge probe in flow-through chamber, waterway, or water body.
    2. Agitate sample by raising and lowering probe.
    3. Allow sufficient time for probe to stabilize to sample temperature and dissolved oxygen.
    4. Read and record the temperature and the value of the dissolved oxygen in mg/L.
    5. Document field analysis data and general observations in the field log book or groundwater sampling sheet.
- Quality Control calibration is performed periodically against the laboratory instrument.

3. Limitations

- At all times, follow safety procedures as defined in the site-specific Health and Safety Plan.
- Collecting measurements from samples in containers will alter the gaseous content of the sample.

- Detection Limit (D.L.) = 0.1 mg/L for 0-10 mg/L, or 0.2 mg/L for 0-20 mg/L; do not record values less than Detection Limit: a zero reading is recorded < 0.1 mg/L or <0.2 mg/L for 0-20 range).
- Freshwater can hold more oxygen than saltwater. The dissolved salt forces dissolved gas out of water thereby lowering the solubility of water. YSI sensors do not measure mg/L, per se, they measure partial pressure of oxygen. So a known relationship between salinity and dissolved oxygen concentration allows for a correction for salinity.

4. References

Standard Methods for the Examination of Water and Wastewater, 18th Edition, Method 4500-H. American Public Health Association (1992).

5. Attachments

None

6. Contact

Brian Conte

STANDARD OPERATING PROCEDURE

QA-001 Equipment Decontamination

1. Objective

This SOP describes methods used for preventing or reducing cross-contamination, and provides general guidelines for sampling equipment decontamination procedures. Preventing or minimizing cross contamination in sampled media and in samples is important for preventing the introduction of error into sampling results and for protecting the health and safety of site personnel. Removing or neutralizing contaminants that have accumulated on sampling equipment ensures protection of personnel from permeating substances, reduces or eliminates transfer of contaminants to clean areas, prevents the mixing of incompatible substances, and minimizes the likelihood of sample cross-contamination.

2. Execution

- Inspect equipment for cleanliness prior to moving onto a site and prior to relocating to each new sampling location. All contractor-provided equipment (augers, rods, spoons, backhoe buckets) shall be decontaminated by steam cleaning **prior to coming on site.**

- Equipment decontamination is a sequential procedure consisting of the following general steps: alconox-solution wash; potable water rinse; methanol wash, and three distilled-water rinses.

- Alconox solution is a mixture of approximately 1 cup of Alconox per 1 gallon of potable water. Alconox solution wash requires scrubbing the equipment with a brush soaked in alconox solution and removing any visible contamination or dirt from the equipment.

- Before advancing each boring, drilling equipment (including augers, casing, rods, and washtub) must be decontaminated by steam cleaning.

- Split-spoon samplers must be decontaminated prior to collecting each sample. The split-spoon decontamination procedure includes: a gross wash and scrub in a bucket of alconox solution; potable water rinse; methanol wash, and three distilled-water rinses.

- Pumps and tubing used for sample collection and well development must be decontaminated by flushing with a minimum of one gallon of potable water; then flushing with a minimum of one pint of methanol and rinsing twice with distilled water.

- For pumps and tubing, perform a final rinse of the sampling equipment with the water being sampled.

3. Limitations

- Do not store the deionized/distilled water in polyethylene bottles, use Nalgene, glass, or Teflon. Polyethylene may leach phthalates.
- Do not attempt to decontaminate string or rope; replace it.
- Due to eye and skin absorption hazards, safety glasses and gloves must be worn when handling decontamination solvents.
- The decontamination procedure may require modification based on site specific conditions and methods used should not interfere with the site-specific chemical analyses. The procedure may also require modification based on state regulations.
- Steam cleaning with potable water is an acceptable decontamination method for drilling equipment (i.e., augers).
- If sampling for metals, the decontamination procedure requires modification to include rinsing with a 1:1 nitric acid and rinsing with deionized water in place of distilled water.
- Dedicated equipment need not be decontaminated beyond initial decontamination prior to field use.

4. References

None

5. Attachments

None

6. Contact

Brian Conte

STANDARD OPERATING PROCEDURE

QA-002 Field and Laboratory Quality Control Procedures

1. Objective

Field quality control (QC) samples are used to monitor the reproducibility and representativeness of the field activities. The QC samples are handled, transported, and analyzed in the same manner as the associated field samples. QC samples may include trip blanks, field blanks, and field duplicates.

2. Execution

2.1. Trip blanks

- Used to monitor possible sources of contamination from transport, storage, inadequate bottle cleaning, or laboratory methodologies.
- Sample containers filled at the laboratory with analyte-free water, are transported to and from the site, and are not opened until time of analysis.
- Trip blanks are stored with the sample containers prior to and after field activities and remain with the collected samples until analyzed.
- Generally, one trip blank per VOC shipment AND when sample shipment is by Fed Ex or other large carrier.

2.2. Field blanks

- Also called equipment blanks, are used to monitor the adequacy of decontamination procedures and possible sources of contamination from inadequate bottle cleaning or laboratory methodologies.
- Field blanks are samples collected by pouring laboratory supplied or distilled or deionized water through a decontaminated piece of field equipment.
- The water is then collected in a sample bottle(s) and stored with the associated field samples and submitted for analysis.
- Generally collected at a frequency of 1/20 samples and when nondedicated sampling equipment is used. Check project-specific work plan and/or quality assurance project plan for required frequency.

2.3. Field duplicates

- Used to evaluate the precision and representativeness of the sampling procedures.
- Field duplicates are two samples collected from the same location using the same procedures. Both samples are submitted to the laboratory as individual samples with different sample identification.

- Field duplicates from groundwater sampling are collected by alternating filling sample containers from the same sampling device. Volatile samples must be collected from the same bailer.
- Soil or sediment field duplicates are collected by homogenizing the sample for all analyses except volatiles. The homogenized sample is then divided into two equal portions and placed in separate sample containers. Field duplicates for volatile analysis are collected at two adjacent sampling locations.
- Each sample is assigned a different sample identification.
- Generally collected at frequency of 1/20 samples. Check project-specific work plan and/or quality assurance project plan for required frequency.

All field QC samples should be labeled in the field and submitted "blind" to the laboratory.

3. Limitations

- At all times, follow safety procedures as defined in the site-specific Health and Safety Plan.
- Trip blanks must never be opened in the field.
- Trip blanks are usually for VOCs only because less volatile compounds are not likely to cross-contaminate other samples by simply being in close proximity.
- Water of documented quality must be used during the collection of field blanks.
- Field duplicates must have different sample identifications.

4. References

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (November 1986), U.S. Environmental Protection Agency Department of Solid Waste, Washington, D.C.

5. Attachments

None

6. Contact

Brian Conte

Title: SOP for State of Connecticut ETPH Method
[Method CTETPH]

Approvals (Signature/Date):

Technical Manager	Date	Health & Safety Manager / Coordinator	Date
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. GCS02703.CT.

Any reference within this document to Severn Trent Laboratories, Inc. or STL, should be understood to refer to TestAmerica Laboratories, Inc. (formerly known as Severn Trent Laboratories, Inc.)

Facility Distribution No. _____ **Distributed To:_____**

Title: SOP for GC/MS Semivolatiles
[Method SW846 8270C]

Approvals (Signature/Date):

Technical Manager	Date	Health & Safety Manager / Coordinator	Date
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. MSS02709.CT.

Any reference within this document to Severn Trent Laboratories, Inc. or STL, should be understood to refer to TestAmerica Laboratories, Inc. (formerly known as Severn Trent Laboratories, Inc.)

Facility Distribution No. _____ Distributed To:_____

1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 The purpose of this SOP is to outline the techniques for determining the presence and concentration of various semivolatile organic target and non-target compounds. The standard 8270C target compounds are listed in Table 1.0. Table 1.1 lists the expanded Appendix IX target compounds applicable to this method. The method used in this procedure is solvent extraction and gas chromatograph/mass spectrometer analysis.

2.2 It is the policy of Testamerica and of the Semivolatiles Group to ensure that we administer contracts and orders for goods and services in a manner that is fully compliant with governmental laws and regulations, as well as the Testamerica Policy Statement on Business Ethics and Conduct.

2.3 The document control number for this SOP is CT-MSS-27, rev. 10.

3.0 TERMS AND DEFINITIONS

3.1 There are many terms and definitions used within the laboratory, which are listed in the latest version of the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 This method employs the technique of solvent extraction, followed by gas chromatograph/mass spectrometer analysis. An aliquot of sample extract is injected onto a gas chromatograph. The fused silica capillary column is then temperature programmed to separate the semi-volatiles organics prior to detection by the mass spectrometer.

4.2 This method is based on SW-846 Method 8270C and Method 8000B. The concentration of the solution used for the initial demonstration of precision and accuracy varies from that listed in the method. The lab uses a concentration of 40 ug/l, and the concentration listed in Table 6 of the method is 100 ug/l. The lab has generated limits based on in house data. These limits are updated on an as needed

basis.

4.3 Some clients (e.g. Army Corps of Engineers) have specific criteria that must be met before proceeding with analysis of samples. The requirements for each specific client are listed in Appendix A of this SOP.

4.4 In the instance where project plans may require lower detection limits, Selected Ion Monitoring (SIM) may analyze certain compounds. Compounds and analysis procedures are noted in Appendix B of this SOP.

4.5 If instrumentation allows, analysis may be performed using simultaneous SIM/SCAN analysis. Compounds and analysis procedures are noted in Appendix C of this SOP.

5.0 INTERFERENCES

5.1 Method interferences may be caused by contaminants in solvents, reagents, and laboratory solvent vapors. This can lead to discrete artifacts and/or elevated baseline in the gas chromatograph. All these materials must be demonstrated to be free from interferences by the running of laboratory reagent blanks.

6.0 HEALTH AND SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

6.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

6.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete

list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 SAMPLE PRESERVATION AND STORAGE

7.1 All sample extracts for semivolatile analysis must be protected from light and refrigerated at 4^0C from the time of extraction until analysis.

7.2 All sample extracts for semivolatile analysis shall be analyzed within 40 days of following extraction.

8.0 APPARATUS AND MATERIALS

8.1 GC/MS/DS System

8.1.1 Gas Chromatograph – Hewlett Packard Model 5890 GC or Agilent 6890 GC, an

analytical system which is temperature programmable with splitless injection and all required accessories including syringes, analytical columns, and gases.

8.1.2 GC Column – Phenomenex ZB-5ms, 30m x 0.25mm ID x 0.25um film thickness, or Restek Corporation RTX5, 30m x 0.25mm ID x 0.5um film thickness columns, or equivalent.

8.1.3 Mass Spectrometer - Hewlett-Packard 5971, 5972, 5973, or 5975 capable of scanning from 35 to 500 AMU's every 1 second or less, utilizing 70 volts (nominal) electron energy in the EI ionization mode, and producing a mass spectrum which meets all the instrument performance criteria when 50 ng or less of DFTPP is injected through the GC inlet. Refer to Table 4.0 for the performance criteria. Any samples analyzed when DFTPP criteria have not been met will require reanalysis.

8.1.4 GC/MS interface - any GC to MS interface that gives acceptable calibration points, at 50 ng or less per injection, for each of the parameters of interest, and achieves all acceptable performance criteria, may be used. GC to MS interfaces constructed of all-glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane.

8.1.5 Data system - Hewlett Packard Chemstation / Enviroquant / Target Software, capable of continuous acquisition and storage, on machine readable media, of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching any data file for ions of a specified mass and plotting such ion abundances versus time or scan number (EICP). Software also allows integrating the abundance in any EICP between specified time limits. Also, software allows for the comparison of sample non-target spectrum against reference library spectra. The most recent release of the NIST/EPA/NIH mass spectral library shall be used as the reference library. The data systems will flag all manual edits with "M" qualifier.

8.1.6 DAT storage unit for long term, off line storage.

8.1.7 Syringes -various volumes.

8.1.8 Micropipets (Wiretrols) - various volumes.

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Stock Standards - Certified standards purchased in ampules from commercial sources of target compound mixes, matrix spike compounds, surrogates, and internal standards. The laboratory utilizes the manufacturers expiration date for the stock

standards. If the manufacturer provides no expiration date, the lab uses 12 months from receipt as the expiration date. The lab uses a 12-month from the preparation date for the expiration of intermediate standards, unless the stocks used to prepare it expire earlier. In that case, the earlier date is used. However, if it appears that the solution has degraded or evaporated, it is replaced sooner.

9.2 Working Standards

9.2.1 Calibration Standard Solutions

Calibration standards at six concentration levels are prepared from the stock solutions. Refer to Table 9 for a list of specific compound concentrations in each level. Each standard contains all the target compounds and surrogates. Continuing calibration standards should be prepared weekly.

9.2.2 Internal Standard (IS) Spiking Solution

A 400 ng/ul IS spiking solution containing the internal standards 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12, is prepared from stock. Add 5 ul of this solution to a 100 ul aliquot of extract for a concentration of 20 ng/ul.

9.2.3 Surrogate (SU), Matrix Spike (MS), and Full Matrix Spike (FMS) Spiking Solution

The acid and base-neutral surrogate, matrix spike, and full matrix spike spiking solutions containing the appropriate compounds in methanol are prepared and utilized by the Extractions group.

9.2.4 DFTPP Tuning Mix

The DFTPP Tuning mix contains DFTPP at 25ug/ml . Alternatively, the continuing calibration standard at 40ug/ml may contain DFTPP, 4'4 DDT, pentachlorophenol, and benzidine to test tuning, breakdown and tailing.

9.2.5 Storage of all working standards for semivolatile analysis must be protected from light and refrigerated at $<10^0$C.

10.0 CALIBRATION

10.1 Calibration Standards

Six initial calibration standards containing all the semivolatile target compounds and SU's are analyzed by injecting 1ul of the calibration standards. The IS compounds are added to all calibration standards at a final concentration of 20 ng/uL.

10.2 The working calibration range of this method is defined by the initial calibration curve, 4 to 80 ng. See table 9.0 for compound specific concentrations. All extracts with target compounds exceeding 80 ng/uL, must be diluted to within the upper half of the calibration range.

10.3 Initial Calibration

The calibration curve is prepared by adding 100 ul of each of the six calibration standards and 5 ul of Internal Standard Solution to a 200 ul conical vial for a total of 105 ul per vial. An initial calibration must be analyzed on each GC/MS system upon installation, whenever corrective action is taken which may affect the initial calibration criteria, or if the continuing calibration criteria cannot be met.

The initial calibration can be performed only after the instrument performance is verified by meeting the DFTPP ion abundance criteria listed in Table 4.0. If less than 12 hours has expired since the tuning compound was injected, after meeting the initial calibration criteria, then samples may be analyzed. Quantitation is based on either the average RF in the curve or a linear regression line. If time does not remain in the 12 hour time period, a continuing calibration must be performed.

A 2nd source ICV is required following an initial calibration curve, with acceptance criteria of 25% difference from the initial calibration standard with a sporadic exceedence of 5% of the analytes. Please refer to Appendix A for ACOE requirements.

The response factors of the system performance check compounds (SPCC's) listed in Table 5.1 must have average relative response factors which are greater than or equal to 0.05, and the continuing calibration check compounds (CCC's) listed in Table 5.0 must have percent relative standard deviations (RSD's) less than or equal to 30.0 percent for the initial calibration to be acceptable. Linear regression or a quadratic equation (must use at least 6 levels for quadratic) may be used if the % RSD does not meet criteria for any compound except a CCC, listed in Table 5.0. The coefficient for the linear regression or quadratic equation must be 0.990 or greater.

A minimum of 5 calibration points are used to calculate the % RSD's. A six-point calibration point is typically run, however, the low point for poorly responding acid compounds may be removed. This removal of the low point is acceptable as long as the reporting limit can still be met by the lowest standard remaining in the curve. A five-point calibration curve is required for all compounds.

There are three options that can be used to determine the acceptability of an initial calibration. They are listed below;

1) If the %RSD of any compound is ≤15%, the average RF from the curve for that compound may be used for quantitation.

2) If the %RSD for one or more analytes is greater than 15%, the mean %RSD for all compounds will be calculated. If the mean %RSD is ≤15%, the average RF may be used for all compounds. Please refer to Appendix A for ACOE requirements.

3) If the mean %RSD is greater than 15%, quantitation from the regression line must be used for those compounds whose %RSD is greater than 15%. Quadratic fit may also be used if there is a 6-point curve. The coefficient must be >= 0.990 for both linear regression and quadratic fit. Please refer to Appendix A for ACOE requirements.

10.4 Continuing Calibration

The continuing calibration can be performed only after the instrument performance is verified by meeting the DFTPP ion abundance criteria listed in Table 4.0. The calibration is verified by injecting a standard containing 40ng/uL of each compound and 50ng or less of DFTPP. The standard must be analyzed every 12 hours.

The response factors of the system performance check compounds (SPCC's) listed in Table 5.1 must have response factors greater than 0.05, and the continuing calibration check compounds (CCC's) listed in Table 5.0 must be less than or equal to 20.0 percent difference (%D) for the continuing calibration to be acceptable. The calculations are listed in Section 13.0. If the continuing calibration does not meet the above criteria the standard must be re-injected. If in the judgment of the analyst routine maintenance will solve the problem it can be performed prior to re-injection of the standard. Examples of routine maintenance include cutting the column, cleaning the injection port, and changing the injection port liner. Repeated failure may require corrective actions and reanalysis of the initial calibration.

11.0 QUALITY CONTROL

11.1 Demonstration of Analyst Capability

11.1.1 Prepare four aliquots of the QC Check Standard at 40 ug/l in reagent water. Process the samples through the whole analytical procedure.

11.1.2 Calculate the average recovery (x) and the standard deviation (s) for each analyte from the four results. Compare the s and x with the criteria generated from the laboratory control charts for each compound for each matrix. The limits are derived from laboratory-generated data, and are updated as needed. If all analytes meet the acceptance criteria, analysis of samples can begin. If any analyte fails, the cause for the failure must be determined and the test must be repeated for that analyte.

11.1.3 The demonstration of analyst capability will be verified on an annual basis.

11.2 Method detection limits (MDL's) for this method will be verified on an annual basis as detailed in the latest version of the corporate SOP on MDL's. The MDL will be verified on each instrument on a quarterly basis by the analysis of an MDL check sample. The MDL check sample is an extracted sample containing each target analyte at a concentration close to the MDL. Each analyte must be detected in order for the instrument to be considered capable of reporting estimated result to the calculated MDL. If an analyte cannot be detected at the given concentration, instrument maintenance should be performed and the MDL check sample re-analyzed. If the compound is still not detected, a new MDL should be prepared and analyzed at a higher concentration. The new MDL should be used in reporting results.

11.3 Method Performance Tests

11.3.1 Prior to initiating any data collection activities it is necessary to establish that a given GC/MS system meets the instrument performance criteria. This is accomplished through the analysis of 50ng or less of decafluorotriphenylphosphine (DFTPP).

11.3.1.1 DFTPP must be analyzed at the start of every 12-hour sequence and can be a component in the 40ng/ul calibration standard.

11.3.1.2 The key ions produced during the analysis of DFTPP and their respective ion abundance criteria are given in Table 4.0. This criteria must be met before any calibration standards, blanks, or samples may be analyzed.

11.3.1.3 If the criteria is not met, the DFTPP must be reanalyzed. Repeated failure shall require the instrument to be manually tuned. After manual tuning, the DFTPP must be re-injected and the abundance criteria must be met before proceeding.

The 12-hour time period for a GC/MS system instrument performance check and standards calibration (initial or continuing calibration criteria) begins at the moment of injection of the DFTPP analysis that is submitted as documentation of

a compliant instrument performance check. The time period ends after 12 hours have elapsed according to the system clock

11.3.2 After the instrument performance criteria is met, an initial calibration is performed, or the initial calibration curve is verified through the analysis of a continuing calibration at 40ng/uL. The continuing calibration criteria must be met before any method blank or sample analyses may proceed.

11.3.3 A method blank spiked with surrogates is extracted with every batch of samples and must be analyzed with the sample extracts after calibration criteria has been met. An acceptable method blank must meet the following criteria:

*Less than or equal to 5X the PQL for the phthalate esters. Refer to Appendix A for ACOE requirements.

*Less than or equal to the PQL for each of the other target compounds. Refer to Appendix A for ACOE requirements.

If the method blank exceeds the above criteria, the analytical system is considered to be out of control. The source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds. All samples analyzed with a method blank that is out of control must be re-extracted and reanalyzed. The problems and solutions must be addressed in the SDG narrative.

When running samples on an instrument and there is not Method blank for that analytical sequence(i.e. dilution reruns), an instrument blank must be run consisting of 100 ul of appropriate solvent with 5 ul of ISTD after initial or continuing calibration criteria and before sample extracts.

11.4 Matrix Spike, Matrix Spike Duplicates and Matrix Spike Blanks

11.4.1 An MS and MSD must be extracted and analyzed for each group of samples of a similar matrix within each case, 20 samples, group of samples of a similar concentration level (soils only), or each 7 calendar day period; whichever is more frequent. An MSB is extracted and analyzed for NYSDEC SW846 work requiring a blank spike in the deliverables.

11.4.2 The lab generated limits for matrix spike compound recovery and relative percent difference (RPD) are given in Table 2.0. These limits are only advisory; therefore, no further action is required if the criteria limits are not achieved. However, frequent failures shall be investigated for possible laboratory generated

error. The NYSDEC MSB has mandatory acceptance criteria. The MSB is evaluated against the matrix specific % Recoveries listed in Table 2.0. If the MSB does not meet criteria, a CAR is written, and the client is informed. The lab will proceed in accordance with the client's instructions.

11.5 Laboratory Control samples (LCS)

11.5.1 A LCS must be extracted with each extraction preparation batch for each group of samples of a similar matrix and concentration level (soils will require separate LCS's if they are extracted by low level or medium level extraction), and must be analyzed with the sample extracts.

11.5.2 The limits for the spike compound recoveries are based on laboratory generated data, and are updated on at least an annual basis. The LCS may have up to 20% of the target list out of acceptance criteria without re-extraction being required. Refer to Appendix A for ACOE criteria.

11.6 Surrogates

11.6.1 The surrogates Phenol-d5, 2,4,6-Tribromophenol, 2-Fluorophenol, Nitrobenzene-d5, Terphenyl-d14, and 2-Fluorobiphenyl are added to each sample, blank, standard, and MS/MSD, prior to extraction. The acid surrogates are added at 75 ug and the base-neutrals at 50 ug for waters and soils.

11.6.2 Surrogate recoveries must be within the lab generated QC limits given in Table 3.0. The QC limits are periodically re-evaluated and updated as necessary. If the recovery for any two acid surrogates (phenol-d5, 2-fluorophenol, and 2,4,6-tribromophenol) or BN surrogates (nitrobenzene-d5, 2-fluorobiphenyl, and terphenyl-d14) are not within the QC limits defined in Table 3.0, or if any one of these surrogate recoveries are below 10%, the following actions are required:

* Check all calculations for accuracy, spiking solutions, and internal standards

* Re-analyze the sample if none of the above steps reveal a problem

* Do not reanalyze dilutions if surrogate recoveries are outside limits

* Never reanalyze the MS or MSD, even if the surrogate recoveries are outside the limits

* If the sample associated with the MS/MSD does not meet specifications, it should be reanalyzed only if the MS/MSD surrogate recoveries are within the limits

If the reanalysis of the sample solves the problem, then only submit the second analysis. If the reanalysis does not solve the problem, then re-extract and reanalyze the sample. If the re-extraction and reanalysis of the sample solves the problem, contact the client by a CAR and find out what they want submitted. If the re-extraction and reanalysis does not solve the problem, then both sets of results may be reported. Re-extraction and reanalysis shall be decided on a project specific basis and may not be requested by the client.

11.6.2.1 Refer to Appendix A for ACOE requirements regarding surrogate recoveries.

11.6.3 If the recovery of the surrogates in a method blank are not acceptable, as defined in section 11.6.2 above, then the method blank and all associated samples must be re-extracted and reanalyzed.

11.7 Internal Standards (IS or ISTD)

11.7.1 Internal standards are added to each sample, blank, standard, and MS/MSD, at 20 ng prior to injection.

11.7.2 The retention times (RT) and extracted ion current profile (EICP) of each IS must be evaluated for all standards immediately after the data acquisition. The IS EICP areas must be monitored and evaluated for each sample, blank, MS, MSD. If the IS EICP changes by more than a factor of 2 (-50% to +100%) from the latest (12 hour) calibration standard, the mass spectrometric system must be inspected for malfunctions, and corrections made as required. If the RT for any IS changes by more than 30 seconds from the latest (12 hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. When corrections are made, reanalysis of the samples analyzed while the system was malfunctioning is necessary. If no system malfunctioning occurred, then document the matrix interference problem via a corrective action report. If the sample associated with the MS/MSD does not meet specifications, it should be reanalyzed only if the MS/MSD IS criteria is within limits.

If the reanalysis of the sample solves the problem, then only submit the second analysis.

11.8 Quality Control Check Points

11.8.1 Analysis Quality Control Approval Report

Specific quality control checkpoints have been established for the analysis of samples which are monitored through a Quality Control Approval Report (QCAR). The

specific check points must be initialed and dated by the analyst to ensure the consistency and accuracy of the data produced. Refer to Figure 1.0 for the QCAR and specific control points covered.

11.9 Analytical Documentation Procedures

11.9.1 Instrument Batches

An instrument batch is created for each analytical sequence to organize all the associated data. Batch designations are of the format:

Xnnnnnn where X = instrument identifier
and nnnnnn = number of batch

(i.e.R051234) Instrument batch numbers are created by combining the last two digits of the year with the first data file number in the batch, creating a unique batch identifier.

11.9.2 Filing System

All active batches are filed chronologically according to instrument. The batches are transferred to file boxes for long-term storage once all the associated data within a batch has been completed.

11.9.3 Data Archiving

The data files and method files on the server are archived on a daily basis by the systems group.

11.9.4 Instrument Run Logs

It is TESTAMERICA's policy that all measurement data be recorded in logbooks or on preprinted log sheets in permanent ink. Transcriptions shall be avoided whenever possible. The record shall reflect the measurement performed and all appropriate details for conclusions related to the measurement. The record shall be signed and dated by the individual performing the measurement on the day the measurement is performed. Corrections must be made by drawing a single line through the error, and initialing and dating the correction. A secondary authorization of the logbook is required and shall be performed by the department's manager or designee.

Each instrument has its own set of bound run logs (see Figure 2.0), which are sequentially number and paginated. Run logs are filed in the laboratory once they have

been filled, for future reference. Each analytical sequence shall be started on a new page of the log and continued on the next page, if necessary. The header information designating the standard codes used shall be completed for each sequence as well as performed maintenance. All standards used are recorded in this field for future traceability. The data file, job number, sample number, dilution factor, analyst's signature, and date are recorded.

11.9.5 Initial data review sheet (BSUM)

The initial data review sheet (IDRS) is a computerized review sheet, which is used to check the key quality control criteria for compliance. The IDRS is used to check that all samples have been analyzed with the required calibration time frame, if the IS's meet RT and EICP area criteria, and if the ID file being utilized was correctly updated. The IDRS is also used as the initial data review tool. Each sample is listed on the sheet and it is either accepted or rejected in the right hand column, by the analyst performing the data review. If reruns are required for dilutions, then the analyst shall indicate the proper dilution required for reanalysis. Refer to Figure 3.0 for an example.

11.9.6 Corrective Action Reports

A corrective action report (CAR) is issued when a problem is encountered during analysis, data reduction or deliverables preparation, data validation, or when any deviations from this SOP occur. The CAR is prepared by the analyst or department manager and is then electronically submitted to the QA/QC department and all affected departments.

11.9.7 Chain of Custody Record

When samples are removed from storage for preparation or analysis they must be signed out utilizing the chain of custody record (COC). The samples shall then be signed back in on the COC upon their return to storage or designated "used" if the sample volume is consumed during the preparation or analysis.

11.9.8 Sample Tracking Record

Samples are tracked on the Semivolatile Tracking Sheet (see Figure 4.0). After the extraction process is complete, the extraction information is entered into Labnet, and the tracking sheet is generated. The tracking sheet is updated after initial data review with the accepted data file for that sample. Samples requiring reanalysis are also tracked with the reason for the reanalysis (i.e. dilution, I.S. confirmation, etc.). If a re-extraction is required, a corrective action report is issued and the sample re-extract tracked accordingly.

12.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

12.1 Instrumental Conditions

12.1.1 Gas Chromatograph (Suggested conditions)

Carrier Gas:	Helium
Flow Rate:	30 cm/sec
Initial Temp.:	40^0C
Initial Hold:	4 min
Ramp Rate 1:	10E/min
Second Temp.:	260^0C
Second Hold:	12 min
Ramp Rate 2:	8^0/min
Final Temp.:	310^0C
Final Hold:	10 min
Injector Temp.:	250^0C
Transfer Temp:	310^0C
Injector:	Grob-type, splitless
Sample Volume:	1 or 2 ul
EPC/EPP	99 mls/min to 40 psi for 0.3 min then 99 mls/min to 7.1 psi (this can be used on instruments equipped with EPC)

12.1.3 Mass Spectrometer

Electron Energy:	70 eV
Mass Range:	35 - 500 amu
Scan Time:	less than 1 sec/scan

The mass spectrometer must be tuned to meet the instrument performance check criteria for 50 ng or less of DFTPP listed in Table 4.0.

12.2 Sample Analysis Procedures

12.2.1 Sample Extract Analysis

Sample extracts are removed from storage (refrigerator #36) and are signed out on the chain of custody form. All sample extracts are signed back in after they returned to

storage.

Make sure all instrumental operating conditions are correctly set and DFTPP and calibration criteria have been met.

In a 200 ul shell vial, load a 100 ul aliquot of sample extract and spike with 5 ul of IS spiking solution.

The prepared extracts are then loaded into the HP auto sampler carousel tray and the sequence is set up in the software to match the injection sequence on the auto sampler tray. If a sample is analyzed which contains target compounds at concentrations greater than the initial calibration upper limit, then the sample must be reanalyzed at an appropriate dilution.

12.3 Qualitative Analysis

12.3.1 Target Compounds

The relative retention time of a target compound must be within +/- 0.06 RRT units of the RRT of the calibration standard for a positive identification. For reference the standard must be analyzed within the same 12 hour time period as the sample. If the sample is analyzed within the same 12 hour time period as the initial calibration, then use the 40 ng standard as the reference. If co-elution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT shall be assigned by using the extracted ion current profiles for ions unique to the component of interest.

In addition, a comparison must be made between the mass spectrum obtained in the sample analysis and the reference mass spectrum for that compound, which was obtained on that specific GC/MS system. The requirements for qualitative verification by comparison of mass spectra are as follows:

All ions present in the reference spectrum at intensity greater than 10% must be present in the sample spectrum.

The relative intensities of the ions above 10% must agree with 20% between the reference and sample spectra.

Ions greater than 10% in the sample spectrum but not present in the reference spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by the above criteria, but in the technical judgment of the analyst, the identification is correct, then the compound shall be reported.

The mass spectral interpretation checklist, refer to Figure 7.0, is used by the mass spectral interpretation specialists to verify the identification of the compounds with recurrent mass spectral interpretation problems. Refer to the checklist for the specific compounds reviewed.

12.3.2 Tentatively Identified Compounds

A library search may be performed upon request for non-target compounds in the sample for purposes of tentative identification. For this purpose, the most recent release of the NIST mass spectral library shall be used. Pesticide and PCB confirmation is also done by tentatively identified compound search if needed to prove the presence of these compounds from a GC analysis.

Up to 20 non-target organic compounds of highest apparent concentration shall be tentatively identified via a forward library search. Only compounds with responses greater than 10% of the closest IS exhibiting no interference are to be searched. Peaks suspected of being aldol-condensation reaction products shall be searched and reported as such, but not counted as part of the 20 most intense non-target compounds. Solvent peaks which may be detected due to an early scan start time shall not be counted as a library search compound.

A tentative identification will be made after a comparison between the mass spectrum obtained in the sample analysis and the library search mass spectra found for that compound. The requirements for tentative verification by comparison of mass spectra are as follows:

Molecular ions present in the reference spectrum should be present in the sample spectrum.

Ions present in the reference spectrum at intensity greater than 10% should be present in the sample spectrum.

The relative intensities of the ions above 10% should agree with 20% between the reference and sample spectra.

Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or co-eluting compounds.

Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible background subtraction by the data system.

If in the technical judgment of the mass spectral interpretation specialist, no valid

tentative identification can be made, the compound shall be reported as unknown. Additional classification shall be made if possible (i.e. unknown hydrocarbon).

12.4 Quantitative Analysis

12.4.1 Target Compounds

Target compounds are quantitated by the internal standard technique. The associated internal standard used is listed in Table 6.0. The EICP area of the quantitation ions of compounds listed in Tables 7.0 and 8.0 are used.

The average relative response factor (RRF) from the curve or the linear regression line from the curve is used to calculate the concentration in the sample. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the SDG narrative. The area of a secondary ion cannot be substituted for the primary ion unless a RRF is calculated using the secondary ion.

When compound concentrations are below the PQL, but the compound meets identification criteria, report the concentration with a "J" qualifier if it is above the MDL.

Water Samples

$$\text{Concentration ug/L} = \frac{(Ax)(Is)(Vt)(Df)}{(Ais)(RRF)(Vo)(Vi)}$$

where,

Ax = area of the compound quantitation ion

Ais = area of IS quantitation ion

Is = IS amount in nanograms

RRF = average RRF from curve

Vo = volume of water extracted in ml's

Vi =volume of extract injected in ul's

Vt =volume of the concentrated extract in ul's

Df =Dilution factor. The dilution factor for the analysis of water samples for semi-volatiles organics by this method is defined as follows:

$$\frac{\text{uL most conc. extract used to make dilution + uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

Soil Samples

$$\text{Concentration ug/Kg (dry weight basis)} = \frac{(Ax)(Is)(Vt)(Df)}{(Ais)(RRF)(Vi)(Ws)(D)}$$

where,

Ax, Is, Ais, Vt, Vi, Df, and RRF are as given for water.

$$D = \frac{100 - \%\ \text{moisture}}{100}$$

Ws = weight of sample extracted in grams

12.4.2 Tentatively Identified Compounds

An estimated concentration for non-target compounds tentatively identified in the sample shall be determined by the internal standard method. For quantitation, the nearest IS free of interferences shall be used.

The equation for calculating concentrations is the same as in 12.4.1. Total area counts from the total ion chromatograms are used for both the IS and compound. A RRF of 1.0 is assumed and the resulting concentration shall be qualified as "J" (estimated) and "N"(presumptive evidence of presence), indicating the quantitative and qualitative uncertainties associated with this non-target compound.

12.4.3 If the on-column concentration of any target compound in any sample extract exceeds the initial calibration range, that sample extract must be diluted, the IS concentration readjusted, and the sample extract reanalyzed. Guidance in performing dilutions, and exceptions to this requirement are as follows:

12.4.3.1 Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range.

12.4.3.2 The dilution factor chosen shall keep the response of the largest analyte peak for a target compound in the upper half of the initial calibration range of the

instrument.

12.4.3.3 Data for more than two analyses shall not be submitted.

12.5 Instrument Maintenance

12.5.1 Preventative Maintenance

All instrumentation is covered by a service contract with an external instrumentation service vendor, or by TESTAMERICA personnel trained in preventative maintenance. Preventative maintenance is performed at scheduled intervals on all equipment according to the manufacturers recommendations. All instrument preventative maintenance is performed according the manufacturers recommended procedures, by trained personnel. All preventative maintenance shall be thoroughly documented in the maintenance log (see Figure 8.0), with a description of the maintenance performed, the date performed, and the personnel performing the maintenance.

12.5.2 Corrective Maintenance Determinants and Procedures

Corrective maintenance is deemed necessary when the analytical system, after reanalysis, cannot meet tune, calibration, or other protocol specific QC criteria. Corrective maintenance may include, but is not limited to, decontamination of the system, source cleaning, replacing the electron multiplier, column replacement or filament replacement. All corrective maintenance is performed according the manufacturers recommended procedures, by trained personnel. All corrective maintenance shall be thoroughly documented in the maintenance log, with a description of the maintenance performed, the date performed, and the personnel performing the maintenance.

12.5.3 Maintenance Authorization

All preventative and corrective maintenance is authorized by the department's manager, or designee. When maintenance is deemed necessary, a service call is placed for all equipment covered under a service contract, by the department's manager, or designee.

12.6 Data System

12.6.1 Data Acquisition and System Operation

Data is acquired from sample analyses using the Hewlett-Packard, ChemStation software. Analytical batches are set up with all the associated sample ID, dilution, and data file information. Automated post-acquisition quantitation performed using Target

software.

12.6.2 Instrument Errors

System errors are logged to a system error file at the time of occurrence. The system manager shall be responsible for checking and providing corrective actions for all major system errors. Minor system errors, such as insufficient disk space, are handled by trained analysts, as necessary.

12.6.3 Manual Integrations and Editing Flags

Manual integrations are required when the automated software does correctly integrate extracted ion current profiles (EICP). Manual integrations are flagged by the data system with the "M" qualifier beside any manually integrated area on the hardcopy quant report. Each analyst shall log into the data processing software with a unique login. The analyst who performed the integration will have their name listed on the Quant report next to the Operator. Secondary review of manual integrations is signed of on the QCAR as the final package is reviewed within the department.

13.0 CALCULATIONS

13.1 Relative Response Factor (RRF)

$$RRF = \frac{(Ax)\,(Cis)}{(Ais)(Cx)}$$

where,

Ax = area of the compound quantitation ion

Ais = area of IS quantitation ion

Cis = IS concentration

Cx = compound concentration

An average RRF is calculated for each compound and surrogate from the initial calibration.

13.2 Percent Relative Standard Deviation (%RSD)

$$\%RSD = \frac{\text{Standard Deviation}}{} \times 100$$

mean

13.3 Percent Difference (%D)

$$\%D = \frac{(\text{Average RRFi}) - (\text{RRFc})}{(\text{Average RRFi})} \times 100$$

where,

Average RRFi = Average RRF from the initial calibration

RRFc = RRF from the continuing calibration standard

13.4 Percent Moisture

$$\%\ \text{moisture} = \frac{\text{g of Wet Sample} - \text{g of Dry Sample}}{\text{g of Wet Sample}} \times 100$$

13.5 Target Compound Concentrations in the extract

13.5.1 The concentration of each identified analyte and surrogate in the extract is calculated from the average RF of the initial calibration or from the linear or quadratic cuve fitted to the initial calibration points.

13.5.2 Average Response factor – If the average of all the RSD's of the response factors in the initial calibration is <= 15%, the average response factor from the initial calibration may be used for quantitation.

$$C_{ex} = \frac{R_x C_{is}}{R_{is} AvgRF}$$

13.5.3 Linear Fit

$$C_{ex} = A + B\,\frac{(R_x C_{is})}{R_{is}}$$

C_{ex} = Concentration in extract, ug/ml
R_x = Response for analyte(area of quantitation ion)
R_{is} = Response for internal standard(area of quantitation ion)
C_{is} = Concentration of internal standard
A = Intercept
B = Slope

The corresponding Target software calculation is as follows:

$$Cex = Cis\left(b + \frac{1}{m1} \times \frac{Rx}{Ris}\right)$$

b = Concentration Ratio Intercept
m1 = Inverse of slope

13.5.4 Quadratic fit

$$Cex = A + B\,(RxCis) + C\,(RxCis)^2$$
$$\qquad\qquad Ris \qquad\qquad Ris$$

C = Curvature

The corresponding Target software calculation is as follows:

$$Cex = Cis\left(b + m1 \times \frac{Rx}{Ris} + m2 \times \left(\frac{Rx}{Ris}\right)^2\right)$$

m1 = First order coefficient
m2 = Curvature(Second order coefficient)

13.6 The concentration in the sample is then calculated.

13.6.1 Aqueous calculation

$$Concentration(ug/L) = \frac{(Cex)(DF)(Vt)}{(Vo)}$$

Vt = Volume of final extract in ul's
Vo = volume of water extracted in ml's
DF = Dilution factor

13.6.2 Sediment/soil, Sludge(on a dry weight basis) and Oil Waste(normally on a wet weight basis:

$$Concentration(ug/Kg) = \frac{(Cex)(DF)(Vt)}{(Ws)(X)}$$

Ws = Weight of Sample extracted or diluted in grams
X = (100-%moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis (moisture factor applied by LIMS)

13.7 Surrogate Percent Recovery

$$\% \text{ Recovery} = \frac{\text{Concentration Found}}{\text{Concentration Spiked}} \times 100$$

13.8 Matrix Spike Recovery / Full Matrix Spike Recovery

$$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

where,

SSR = spiked sample result

SR = sample result

SA = spike added

13.9 Relative Percent Difference

$$RPDc = \frac{\text{Absolute } (Cms - Cmsd)}{(1/2)(Cms+Cmsd)} \times 100\%$$

where,

Cms = Concentration of matrix spike

Cmsd= Concentration of matrix spike duplicate

The absolute value of the concentration difference is used in the above equation.

13.9.1 Adjusted Estimated Quantitation Limit for Samples

$$\text{Adjusted EQL} = \frac{(EQL) \times Df}{D}$$

where,

$$D = \frac{100 - \% \text{ Moisture}}{100}$$

Df =Dilution Factor

14.0 ACCEPTANCE OF DATA

14.1 Method Blank

The method blank must contain less than or equal to the PQL of the target compounds, except the phthalate esters, which must be less than or equal to five times the PQL. Refer to Appendix A for ACOE criteria.

14.2 Surrogates (SU)

Refer to section 11.6 for surrogate acceptance criteria and corrective actions.

14.3 Instrument Performance Check

The criteria for DFTPP is listed in Table 4.0 and in section 11.3.1.

14.4 Internal Standards

The IS acceptance criteria is described in section 11.7.

14.5 Matrix Spike/Matrix Spike Duplicate

The MS/MSD acceptance criteria is described in section 11.4.

15.0 REPORTING OF RESULTS

15.1 General Information

The following samples suffixes are used:

MS = Matrix Spike
MSD = Matrix Spike Duplicate
RE = Reanalysis or Re-extract
DL = Secondary Dilution
MSB = Matrix Spike Blank (used as a prefix)
FMS = Full Matrix Spike

.

15.2 Analysis Data Sheet

All results are reported uncorrected for blank contaminants and to one significant figure if the concentration is less than 10, and two significant figures if the concentration is greater than 10. Values below the MDL are not reported.

Data Reporting Qualifiers are as follows:

U = Analyzed for, but not detected.
J = Estimated value; used for all TIC concentrations and all targets with are less than the EQL adjusted for extraction volumes/% M.
B = Compound detected in the blank as well as in sample.

E = Concentration exceeds the working range of initial calibration.
D = All compounds detected in analysis at a secondary dilution.
N = Presumptive evidence of a compound; qualifiers at TIC results which have been tentatively identified with a CAS #.

Other qualifiers are defined in the report package.

15.3 Case Narrative

A case narrative is produced for every SDG. Any problems encountered during analysis, data reduction, or any deviation from standard operating procedures will be noted here.

15.4 Forms

Necessary forms will be determined by the level of deliverables.

16.0 SUPPLEMENTAL DOCUMENTS

16.1 The analyst is referred to other departments SOP's for additional information concerning sample storage, sample removal, sample tracking and other information not included in this SOP.

17.0 POLLUTION PREVENTION

17.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

17.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

17.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

17.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

17.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

17.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

18.0 WASTE MANAGEMENT

18.1 All waste shall be managed in accordance with all state and federal requirements. See the TESTAMERICA-CT RCRA Contingency Plan.

18.2 All personnel who handle or generate waste must be trained within six months of employment in proper waste handling and requirements.

19.0 SUPPLEMENTAL DOCUMENTS

19.1 There is a variety of SOP's available to the analyst that document how to perform ancillary tasks that are not detailed in this SOP.

19.2 SOP for Control Limits

20.0 REFERENCES

20.1 USEPA SW-846, Method 8270C

21.0 SUBSTANTIVE REVISIONS

21.1 Original Issue - 01/06/98.

21.2 Changed AEN to STL, and minor editorial changes - 2/10/99.

21.3 Added appendix for Army Corps of Engineers specific requirements - 3/16/99.

21.4 Added new Section 3 - Terms and Definitions, Section 16 - Pollution Prevention, Section 17 - Waste Management, and Section 18 - Supplemental documents. Renumbered sections as appropriate, and minor editorial changes - 10/10/99.

21.5 Minor editorial changes – 05/15/01.

21.6 Updated to include minor procedural changes due to the implementation of Target and labnet software. Expanded the standards expiration dates section, and made minor procedural and editorial changes, including lowering the concentration used for the acid surrogates. Incorporated lab generated surrogate and matrix spike control limits into SOP, and revised Figures to current versions – 10/15/02.

21.7 Updated Section 6.0 for health and safety. Updated Section 10.0 to include 6 point calibration curves, linear regression and quadratic coefficient criteria. Updated 11.5.2 LCS criteria for PAH list. Updated references to tables and figures.

21.8 Section 9.2.4 added. Removed misleading statement from section 10.3 that 'no continuing calibration standard was required'. Added 2nd source ICV to section 10.3. Added linear regression and quadratic coefficient criteria. Referenced Appendix A for ACOE criteria. Section 11.1 and 11.1.3 reworded to analyst capability. Section 11.2 – clarified procedure of the quarterly check of MDL check samples. 11.3.3 added reference to Appendix A for ACOE criteria. Section 11.5.1 soils only statement clarified.. Section 11.5.2 added reference to Appendix A for ACOE criteria. Added Section 11.6.2.1 for reference to Appendix A for ACOE criteria. Section 12.3.2 added a TIC requirement. Section 12.4.1 removed manual integration signature requirement. Section 13.5 and 13.6 added calculations for a target compound. Section 14.1 added reference to Appendix A for ACOE criteria. Section 13.8 – changed RPD to calculate from concentration rather than recovery. Table 1 – clarified dry weight notation. Section 9.2.5 added. Updates to Appendix A – Changed initial calibration criteria. Added linear regression and quadratic fix requirements. Changed continuing calibration criteria. Section 4.1 changed MB criteria. Added Section 5 for LCS criteria. Added Section 6 for Surrogate criteria. Changed ACOE requirements to APPENDIX A. Changed SIM analysis to APPENDIX B.

21.9 Revised section 9.2 to include weekly preparation of the continuing calibration standard. 6-24-05.

21.10 Added section 4.5 to include reference to sim/scan analysis. Added 5975 to Section 8.1.3. Std concentrations changed, referenced table 9.0 in section 9.2.1 and 10.2. Removed reference to 2ul injection from sect 10.1. Clarified use of linear regression with CCC's in Section 10.3. Updated continuing calibration concentration in section 10.4. Changed CAR to electronic submittal in section 11.9.6. 12/15/05.

21.11 Added Phenomenex column information to section 8.1.2. Added the need to run an instrument blank before sample extracts if no MB is run to section 11.3.3. Added need to log minor maintenance performed in the header section of run logs

to section 11.9.4. 05/10/07

21.12 Section 11.5.2 – LCS changed from 12 out to 20% of target list can be out of criteria 5/10/07.

21.13 Added Organophosporous Pesticide, Zeneca Pesticide, and Golder compounds to Appendix A for target compound list table

21.14 Added new TestAmerica SOP header and control number, changed company name, 01/10/08.

APPENDIX A

Army Corps of Engineers Specific Requirements (Rev. 11/99)

1.0 Daily Performance Test

1.1 On a daily basis the inertness of the injection port and the GC column performance will be evaluated by the analysis of a standard containing 50 ng of 4,4'-DDT, pentachlorophenol, and benzidine. These compounds can either be added to the separate DFTPP tuning standard, or to the 50 ng. continuing calibration verification standard.

1.2 The DDT must be evaluated for degradation to DDD and DDE. The combined breakdown cannot exceed 20%. The breakdown determination option included in the Enviroquant software package may be used to determine the breakdown. Print the report for each breakdown pair for inclusion in the batch folder.

1.3 Determine the tailing factor for pentachlorophenol and benzidine using the tailing option included in the Enviriquant software package. The pentachlorophenol tailing factor must be less than 5, and the benzidine tailing factor must be less than 3. Print the tailing factor determination for inclusion in the batch folder.

1.4 Additionally, benzidine and pentachlorophenol must be present at their normal response.

2.0 Initial Calibration

2.1 After the initial calibration is run, examine the %RSD for all the target compounds. If the %RSD is $\leq$15%, the average RF may be used for quantitation.

There are two options that can be used to determine the acceptability of an initial calibration. They are listed below;

1) If the %RSD of any compound is $\leq$15%, the average RF from the curve for that compound may be used for quantitation.

2) If the %RSD of any analyte is greater than 15%, quantitation from the regression line must be used for those compounds whose %RSD is greater than 15%. Quadratic fit may also be used if there is a 6 point curve. The coefficient must be >= 0.995 for linear regression and >= 0.990 for a quadratic fit.

2.2 If after a reasonable attempt has been made to correct any problems with the injection

port, column or source, any analyte fails to meet the above requirements, outliers will be noted in the case narrative.

2.3 A 2nd source ICV is required following an initial calibration curve, with acceptance criteria of 25% difference from the initial calibration standard.

3.0 Continuing Calibration Verification

3.1 All target compounds should not exceed 20% Difference from the initial calibration in order to proceed with the daily analysis of samples.

4.0 Method Blank Acceptance Criteria

4.1 Method blanks must meet the following criteria:

Less than one half the PQL for all target compounds except the phthalate isomers

Less than the PQL for common contaminants such as the phthalate isomers

5.0 LCS Acceptance Criteria

5.1 The acceptance criteria for the LCS will comply with the requirements in DoD QSM. The laboratory shall maintain in-house statistically derived control limits to demonstrate its performance. The maximum allowed number of sporadic marginal exceedences will be no more than 5% of the total number of analytes spiked in the LCS.

6.0 Surrogate Acceptance Criteria

6.1 Surrogate recoveries must be within ACOE Table D-3 of Appendix DoD-D of the DoD QSM. If any surrogate fails to meet this criteria, the sample shall be re-extracted within hold. If matrix proves the result of surrogate failure, only one set of results shall be reported. If the sample is outside of extract hold, the client shall be notified. If re-extraction is outside of hold and sample matrix proves or disproves the result of surrogate failure, both sets of results will be reported.

TABLE 1.0
TARGET COMPOUND LIST (TCL) AND PRACTICAL QUANTITATION LIMITS (PQL)

Semi-Volatile Organics	Quantitation Limits*	
	Water (ug/L)	Low Soil (ug/Kg)
Phenol	10	330
bis(2-Chloroethyl)ether	10	330
2-Chlorophenol	10	330
1,2-Dichlorobenzene	10	330
1,4-Dichlorobenzene	10	330
Benzyl Alcohol	10	330
1,3-Dichlorobenzene	10	330
2-Methylphenol	10	330
2,2'-oxybis(1-Chloropropane)#	10	330
4-Methylphenol	10	330
N-Nitroso-di-n-propylamine	10	330
Hexachloroethane	10	330
Nitrobenzene	10	330
Isophorone	10	330
2-Nitrophenol	10	330
2,4-Dimethylphenol	10	330
Benzoic Acid	50	1,700
bis(2-Chloroethoxy)methane	10	330
2,4-Dichlorophenol	10	330
1,2,4-Trichlorobenzene	10	330
Naphthalene	10	330
4-Chloroaniline	10	330
Hexachlorobutadiene	10	330
4-Chloro-3-methylphenol	10	330
2-Methylnaphthalene	10	330
Hexachlorocyclopentadiene	10	330
2,4,6-Trichlorophenol	10	330
2,4,5-Trichlorophenol	50	1700
2-Chloronaphthalene	10	330
2-Nitroaniline	50	1700
Dimethylphthalate	10	330

Semi-Volatile Organics	Quantitation Limits*	
	Water (ug/L)	Low Soil (ug/Kg)
2,4-Dinitrotoluene	10	330
Diethylphthalate	10	330
4-Chlorophenyl-phenylether	10	330
Fluorene	10	330
4-Nitroaniline	20	670
4,6-Dinitro-2-methylphenol	50	1667
N-nitrosodiphenylamine	10	330
4-Bromophenyl-phenylether	10	330
Hexachlorobenzene	10	330
Pentachlorophenol	50	1667
Phenanthrene	10	330
Anthracene	10	330
Di-n-butylphthalate	10	330
Fluoranthene	10	330
Pyrene	10	330
Butylbenzylphthalate	10	330
3,3'-Dichlorobenzidine	10	330
Benzo(a)anthracene	10	330
Chrysene	10	330
bis(2-Ethylhexyl)phthalate	10	330
Di-n-octylphthalate	10	330
Benzo(b)fluoranthene	10	330
benzo(k)fluoranthene	10	330
Benzo(a)pyrene	10	330
Indeno(1,2,3-cd)pyrene	10	330
Dibenzo(a,h)anthracene	10	330
Benzo(g,h,i)perylene	10	330
Acenaphthylene	10	330
3-Nitroaniline	50	1700
Acenaphthene	10	330
2,4-Dinitrophenol	50	1700
4-Nitrophenol	50	1700

Dibenzofuran	10	330

*When a sample is reported, quantitation limits are based on wet weight. This table is to indicate the starting quantitation limit. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

#Previously known as bis(2-chloroisopropyl)ether.

TABLE 1.1
APPENDIX IX PRACTICAL QUANTITATION LIMITS

Semi-Volatile Organics	Quantitation Limits	
	Water (ug/L)	Low Soil (ug/Kg)
1-Naphthylamine	50	1700
1,2,4-Trichlorobenzene	10	330
1,2,4,5-Tetrachlorobenzene	10	330
1,3,5-Trinitrobenzene	50	1,700
1,4-Naphthoquinone	100	3,300
2-Acetylaminofluorene	10	330
2-Chloronaphthalene	10	330
2-Chlorophenol	10	330
2-Methylnaphthalene	10	330
2-Naphthylamine	50	1,700
2,3,4,6-Tetrachlorophenol	50	1,700
2,4-Dichlorophenol	10	330
2,4-Dimethylphenol	10	330
2,4-Dinitrophenol	50	1,700
2,4-Dinitrotoluene	10	330
2,4,5-Trichlorophenol	50	1,700
2,4,6-Trichlorophenol	10	330
2,6-Dichlorophenol	50	1,700
2,6-Dinitroluene	10	330
3-Methylcholanthrene	10	330
3,3'-Dichlorobenzidine	20	670
3,3'-Dimethylbenzidine	50	1,700
4-Aminobiphenyl	10	330
4-Bromophenyl phenylether	10	330
4-Chlorophenyl phenylether	10	330
4,6-Dinitro-o-methylphenol	50	1,700
4-Nitroquinoline-1-oxide	50	1,700
5-Nitro-o-toluidine	10	330
7,12-Dimethylbenz[a]anthracene	50	1,700
Acenaphthene	10	330

Semi-Volatile Organics	Quantitation Limits	
	Water (ug/L)	Low Soil (ug/Kg)
Acenaphthylene	10	330
Acetophenone	10	330
alpha,alpha-Dimethylphenethylamine	100	3,300
Aniline	10	330
Anthracene	10	330
Aramite	100	3,300
Benzo[a]anthracene;Benzanthracene	10	330
Benzo[a]pyrene	10	330
Benzo[b]fluoranthene	10	330
Benzo[g,h,i]perylene	10	330
Benzo[k]fluoranthene	10	330
Benzyl alcohol	10	330
bis(2-Chloroethoxy)methane	10	330
bis(2-Chloroethyl)ether	10	330
bis(2-Ethylhexyl)phthalate	10	330
bis(2-Chloroisopropyl)ether	10	330
Butylbenzylphthalate	10	330
Chrysene	10	330
Di-n-butylphthalate	10	330
Di-n-octylphthalate	10	330
Diallate	10	330
Dibenzofuran	10	330
Dibenz[a,h]anthracene	10	330
Diethylphthalate	10	330
Dimethylphthalate	10	330
Dinoseb	10	330
Diphenylamine	10	330
Ethylmethanesulfonate	10	330
Fluoranthene	10	330
Fluorene	10	330
Hexachloroethane	10	330
Hexachlorobenzene	10	330

Hexachlorobutadiene	10	330
Hexachlorocyclopentadiene	10	330
Hexachlorophene	IND	IND
Hexachloropropene	10	330
Indeno(1,2,3-cd)pyrene	10	330
Isophorone	10	330
Isosafrole	10	330
m-Methylphenol(3&4)total	10	330

TABLE 1.1 (continued)
APPENDIX IX PRACTICAL QUANTITATION LIMITS

Semi-Volatile Organics	Quantitation Limits	
	Water (ug/L)	Low Soil (ug/Kg)
m-Dichlorobenzene (1,3)	10	330
m-Dinitrobenzene	10	330
m-Nitroaniline (3-)	50	1,700
Methapyrilene	10	330
Methylmethanesulfate	10	330
N-Nitroso-di-n-butylamine	10	330
N-Nitroso-diethylamine	20	670
N-Nitroso-dimethylamine	10	330
N-Nitrosodiphenylamine	10	330
N-Nitroso-di-n-propylamine	10	330
N-Nitroso-methylethylamine	10	330
N-Nitroso-morpholine	10	330
N-Nitroso-pyrrolidine	10	330
Naphthalene	10	330
Nitrobenzene	10	330
o-Toluidine	10	330
o-Dichlorobenzene (1.2-)	10	330
o-Nitroaniline (2-)	50	1,700
o-Nitrophenol (2)	10	330
o-Methylphenol (2)	10	330
p-Phenylenediamine	IND	IND
p-Nitroaniline (4-)	50	1,700
p-(Dimethylamino)azobenzene	10	330
p-Nitrophenol (4-)	50	1,700
p-Dichlorobenzene (1,4-)	10	330
p-Chloro-m-methylphenol	10	330
p-Chloroaniline (4-)	10	330
Pentachlorobenzene	10	330
Pentachloronitrobenzene	10	330
Pentachlorophenol	50	1,700

Phenacetin	10	330
Phenanthrene	10	330
Phenol	10	330
Pronamide	10	330
Pyridine	25	830
Pyrene	10	330
Safrole	10	330

TABLE 1.2
ORGANOPHOSPOROUS PESTICIDES PRACTICAL QUANTITATION LIMITS

Semi-Volatile Organics	Quantitation Limits	
	Water (ug/L)	Low Soil (ug/Kg)
2-Picoline	10	330
o,o,o-TEPA	10	330
Thionazin	10	330
Sulfotep	10	330
Phorate	10	330
Dimethoate	10	330
Disulfoton	10	330
Methyl parathion	10	330
Parathion	10	330
Famphur	10	330

TABLE 1.3
GOLDER PRACTICAL QUANTITATION LIMITS

Semi-Volatile Organics	Quantitation Limits	
	Water (ug/L)	Low Soil (ug/Kg)
Indane	10	330
1-Propynyl Benzene	10	330
m-Toluidine	10	330
2-Chloroaniline	10	330
2-Nitrotoluene	10	330
3-Chloroaniline	10	330
Benzo (b) Thiopene	10	330
1-Chloro-2-nitrobenzene	10	330
Dichloran	10	330
Diphenamid	10	330

TABLE 1.2
TCLP PRACTICAL QUANTITATION LIMITS

Semi-Volatile Organics	Estimated Quantitation Limits
1,4-Dichlorobenzene	20
Hexachloroethane	20
Nitrobenzene	20
Hexachlorobutadiene	20
2,4,6-Trichlorophenol	20
2,4,5-Trichlorophenol	100
2,4-Dinitrotoluene	20
Hexachlorobenzene	20
Pentachlorophenol	100
2-Methylphenol	20
3&4-Methylphenol	20
Pyridine	40

TABLE 2.0
MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

Compound	% Recovery Water	RPD Water	% Recovery Soil	RPD Soil

QC limits are updated in the Laboratory Information Management System.

TABLE 3.0
SURROGATE RECOVERY LIMITS

QC limits are updated in the Laboratory Information Management System.

TABLE 4.0
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA FOR
QUADRAPOLE MASS SPECTROMETERS

Mass	Ion Abundance Criteria
51	30.0 - 60 percent of mass 198
68	Less than 2.0 percent of mass 69
69	Greater than 1.00% and less than 100.00% of mass 198
70	Less than 2.0 percent of mass 69
127	40-60 percent of mass 198
197	Less than 1.0 percent of mass 198
198	Base peak, 100 percent relative abundance (see note)
199	5.0-9.0 percent of mass 198
275	10.0 - 30.0 percent of mass 198
365	Greater than 1 percent of mass 198
441	Present, but less than mass 443
442	40.0 - 100.0 percent of mass 198
443	17 - 23 percent of mass 442

TABLE 5.0

Calibration Check Compounds (ccc)	
Phenol	Acenaphthene
1,4-Dichlorobenzene	N-Nitrosodiphenylamine
2-Nitrophenol	Pentachlorophenol
2,4-Dichlorophenol	Fluoranthene
Hexachlorobutadiene	Di-n-octylphthalate
4-Chloro-3-methylphenol	Benzo(a)pyrene
2,4,6-Trichlorophenol	

TABLE 5.1

System Performance Check Compounds (spcc)	
N-Nitroso-di-n-propylamine	Hexachlorocyclopentadiene
2,4-Dinitrophenol	4-Nitrophenol

TABLE 6.0
SEMI-VOLATILE INTERNAL STANDARDS WITH CORRESPONDING TARGET COMPOUNDS AND SURROGATES ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d_4	**Naphthalene-d_8**	**Acenaphthene-d_{10}**	**Phenanthrene-d_{10}**	**Chrysene-d_{12}**	**Perylene-d_{12}**
Phenol	Nitrobenzene	Hexachlorocyclopentadiene	4,6-Dinitro-2-methylphenol	Pyrene	Di-n-octylphthalate
bis(2-Chloroethyl)ether	Isophorone	2,4,6-Trichlorophenol	N-Nitrosodiphenylamine	Butylbenzylphthalate	Benzo(b)fluoranthene
2-Chlorophenol	2-Nitrophenol	2-Chloronaphthalene*	4-Bromophenyl phenyl ether	3,3'-Dichlorobenzidine	Benzo(k)fluoranthene
1,3-Dichlorobenzene	2,4-Dimethyl-phenol	2-Nitroaniline	Hexachlorobenzene	Benzo(a)anthracene	Benzo(a)pyrene
1,4-Dichlorobenzene	bis(2-Chloroethoxy)methane	Dimethylphthalate	Pentachlorophenol	bis(2-Ethylhexyl)phthalate	Indeno(1,2,3-cd)pyrene
1,2-Dichlorobenzene	2,4-Dichlorophenol	Acenaphthylene	Phenanthrene	Chrysene	Dibenz(a,h)anthracene
2-Methylphenol	1,2,4-Trichlorobenzene	3-Nitroaniline	Anthracene	Terphenyl-d_{14} (surr)	Benzo(g,h,i)perylene
2,2'-oxybis-(1-Chloropropane)	Naphthalene	Acenaphthene	Di-n-butylphthalate		
4-Methylphenol	4-Chloroaniline	2,4-Dinitrophenol	Fluoranthene		
N-Nitroso-di-n-propylamine	Hexachlorobutadiene	4-Nitrophenol			
Hexachloroethane	4-Chloro-3-methylphenol	Dibenzofuran			
2-Fluorophenol (surr)	2-Methylnaphthalene	2,4-Dinitrotoluene			
Phenol-d_5 (surr)	Nitrobenzene-d_5 (surr)	2,6-Dinitrotoluene			
		2,4,5-Trichlorophenol			
		Diethylphthalate			
		4-Chlorophenyl phenyl ether			
		Fluorene			
		4-Nitroaniline			
		2-Fluorobiphenyl (surr)			
		2,4,6-Tribromophenol (surr)			

TABLE 7.0
CHARACTERISTIC IONS FOR INTERNAL STANDARDS
FOR SEMI-VOLATILE COMPOUNDS

Internal Standards	Primary Ion	Secondary Ions
1,4-Dichlorobenzene-d_4	152	115
Naphthalene-d_8	136	68
Acenaphthene-d_{10}	164	162, 160
Phenanthrene-d_{10}	188	94, 80
Chrysene-d_{12}	240	120, 236
Perylene-d_{12}	264	260, 265

TABLE 8.0
CHARACTERISTIC IONS FOR SEMI-VOLATILE TARGET COMPOUNDS AND SURROGATES

Parameter	Primary Ion	Secondary Ion(s)
Phenol	94	65, 66
bis(2-Chloroethyl)ether	93	63, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene	146	148, 113
1,2-Dichlorobenzene	146	148, 113
2-Methylphenol	108	107
2,2'-oxybis(1-Chloropropane)	45	77, 79
4-Methylphenol	108	107
N-Nitroso-di-propylamine	70	42, 101, 130
Hexachloroethane	117	201, 199
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	65, 109
2,4-Dimethylphenol	107	121, 122
bis(2-Chloroethoxy)methane	93	95, 123
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129, 127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223, 227
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235, 272
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
2-Chloronaphthalene	162	164, 127
2-Nitroaniline	65	92, 138
Dimethylphthalate	163	194, 164
Acenaphthylene	152	151, 153
3-Nitroaniline	138	108, 92
Acenaphthene	153	152, 154

2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	109	139, 65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63, 182
2,6-Dinitrotoluene	165	89, 121
Diethylphthalate	149	177, 150
4-Chlorophenyl-phenylether	204	206, 141
Fluorene	166	165, 167
4-Nitroaniline	138	92, 108
4,6-Dinitro-2-methylphenol	198	182, 77

TABLE 8.0 (continued)
CHARACTERISTIC IONS FOR SEMI-VOLATILE TARGET COMPOUNDS AND SURROGATES

Parameter	Primary Ion	Secondary Ion(s)
N-Nitrosodiphenylamine	169	168, 167
4-Bromophenyl-phenylether	248	250, 141
Hexachlorobenzene	284	142, 249
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Anthracene	178	179, 176
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 100
Pyrene	202	101, 100
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126
Benzo(a)anthracene	228	229, 226
bis(2-Ethylhexyl)phthalate	149	167, 279
Chrysene	228	226, 229
Di-n-octylphthalate	149	---
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(a)pyrene	252	253, 125
Indeno(1,2,3-cd)pyrene	276	138, 227
Dibenz(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277

SURROGATES

Parameter	Primary Ion	Secondary Ion(s)
Phenol-d_5	99	42, 71
2-Fluorophenol	112	64
2,4,6-Tribromophenol	330	332, 141
Nitrobenzene-d_5	82	128, 54
2-Fluorobiphenyl	172	171
Terphenyl	244	122, 212

FIGURE 7.0

MASS SPECTRAL INTERPRETATION CHECKLIST

The following compounds are commonly mis-identified due to mass spectral interpretation errors. Please review the mass spectrum and retention times of the following compounds for the described problems when making identification decisions.

COMPOUND	m/z	RT(*)	Potential Identification Problem
1,4-dichlorobenzene-d4	152	6.40	misidentified as 1,2-dichlorobenzene-d4
1,2-dichlorobenzene-d4	152	6.67	misidentified as 1,4-dichlorobenzene-d4
aniline(TIC)	93	5.99	misidentified as bis(2-chloroethyl)ether
bis(2-chloroethyl)ether	93	6.05	misidentified as aniline
1,3-dichlorobenzene	146	6.35	misidentified as 1,4 or 1,2-isomer
1,4-dichlorobenzene	146	6.42	misidentified as 1,3 or 1,2-isomer
1,2-dichlorobenzene	146	6.69	misidentified as 1,3 or 1,4-isomer
4-chlorophenol (TIC)	128	8.22	misidentified as naphthalene
naphthalene	128	8.22	misidentified, actually 4-chlorophenol)
benzyl alcohol	108	6.62	not identified, coelutes with surrogate 1,2-dichlorobenzene-d4 or misidentified with 2-methylphenol or 4-methylphenol
2-methylphenol	108	6.79	misidentified as benzyl alcohol or 4-methylphenol
4-methylphenol	108	6.98	misidentified as benzyl alcohol or 2-methylphenol
2,4,6-trichlorophenol	196	9.42	misidentified as 2,4,5-isomer
2,4,5-trichlorophenol	196	9.46	misidentified as 2,4,6-isomer
2-nitroaniline	138	9.76	misidentified as 3-nitroaniline or 4-nitroaniline
3-nitroaniline	138	10.17	misidentified as 2-nitroaniline or 4-nitroaniline
4-nitroaniline	138	10.74	misidentified as 2-nitroaniline or 3-nitroaniline
phenanthrene	178	11.54	misidentified as anthracene
anthracene	178	11.58	misidentified as phenanthrene
flouranthene	202	12.54	misidentified as pyrene
pyrene	202	12.74	misidentified as fluoranthene
benzo(a)anthracene	228	13.72	misidentified as chrysene
chrysene	228	13.76	misidentified as benzo(a)anthracene
benzo(b)fluoranthene	252	14.84	misidentified as benzo(k)fluoranthene or benzo(a)pyrene
benzo(k)flouranthene	252	14.86	misidentified as benzo(b)fluoranthene or benzo(a)pyrene
benzo(a)pyrene)	252	15.24	misidentified as benzo(b)fluoranthene or benzo(k)flouranthene
indeno(1,2,3-cd)pyrene	276	16.94	misidentified as dibenz(a,h)anthracene or benzo(ghi)perlyene
benzo(ghi)perylene	276	17.43	misidentified as indeno(1,2,3-cd)pyrene
dimethylphthalate	149	9.95	misidentified as another phthalate
diethylphthalate	149	10.61	misidentified as another phthalate
di-n-butylphthalate	149	11.99	misidentified as another phthalate
butylbenzylphthalate	149	13.23	misidentified as another phthalate
bis-2-ethylhexylphthalate	149	13.68	misidentified as another phthalate
di-n-octylphthalate	149	14.24	misidentified as another phthalate

* - The retention times listed will vary according to column length but the elution order will not.

TABLES

FIGURES

APPENDIX B

Semivolatile Sim Analysis

1. Prior to the start of any analysis, the instrument must meet the ion abundance criteria for 50ng or less of DFTPP injected in the scan mode. Additionally, the Electron Multiplier voltage in the DFTPP method must be identical to the voltage in the SIM method. Standards and/or samples can be analyzed for twelve hours from the injection of a compliant DFTPP Tune. The last injection must occur within a twelve hour period, but the run can end outside of the twelve hours.

2. The SIM method should contain a primary ion and at least one (but preferably two) confirmatory ions for each compound. There are some compounds for which only a primary ion is practical, such as Benzidine. The ions for the compounds will be placed in groups based on retention times of the compounds. The total cycle time for the ions in a group should not exceed one second. The dwell time for any ion in a group is typically 30-100msec.

3. The initial calibration curve will consist of at least 5 points. The typical calibration curve consists of the following levels: 0.1ng/ul, 0.25ng/ul, 0.5ng/ul, 1.25ng/ul, 2.5ng/ul, 5.0ng/ul, and 10ng/ul. The continuing calibration level is 0.5ng/ul. There are known problems with pentachlorophenol responding poorly at low concentrations, and that compound may be reported using a four point initial calibration. However, if that occurs, it must be noted in the case narrative. The internal standards used are the same as those for the full SCAN analysis however the concentration added is at 0.5ng/ul.

4. The STL-CT SIM reporting list *with example retention times and suggested groupings) is presented below:

Compound	Ions	RT	Quant IS
Group 1			
Phenol	94,65,66		1
Aniline	93,66		1
2-Chlorophenol	128,64,130		1
1,4-dichlorobenzene-d4(I1)	152,115	3.02	n/a
Group 2			
2-Methylphenol	108,107		
4-Methylphenol	108,107		
Hexachloroethane	117,201,199		1
2,4-dichlorophenol	162,164,98		
Group 3			
Naphthalene-d8(I2)	136,68	4.25	n/a
Naphthalene	128,129,127	4.27	2
Hexachlorobutadiene	225,223,227	4.40	
2-Methylnaphthalene	142,141	4.97	2
Group 4			
Acenaphthylene	152,151,153	5.89	3
Acenapthene-d10(I3)	164,162,160	6.04	n/a
Acenapthene	153,154,1542	6.07	3
Group 5			
Fluorene-d10(S)	176,88,177	6.59	3
Fluorene	166,165,167	6.62	3
Hexachlorobenzene	284,142,249	7.17	
Group 6			
Pentachlorophenol	266,264,268	7.40	4
Group 7			
Phenathrene-d10(I4)	188,94,189	7.58	n/a
Phenathrene	178,179,176	7.61	4
Anthracene	178,176,179	7.66	4

Group 8			
Fluoranthene	202,101,100	8.85	4
Benzidine	184		
Pyrene-d10(S)	212,106,213	9.057	4
Pyrene	202,101,100	9.06	5
3,3'-Dimethylbenzidine	212,196		
Group 9			
3,3'-Dichlorobenzidine	252,254,126	10.33	
Bis(2-ethylhexyl)phthalate	149,167,279	10.41	5
Chrysene-d12(I5)	240,120,236	10.35	n/a
Benzo(a)anthracene	228,229,226	10.33	5
Chrysene	228,229,226	10.37	5
Group 10			
Benzo(b)fluoranthene	252,253,125	11.38	6
Benzo(k)fluoranthene	252,253,125	11.41	6
Benzo(a)pyrene	252,253,125	11.70	6
Perylene-d12(I6)	264,260,265	11.76	n/a
Group 11			
Indeno(1,2,3-cd)pyrene	276,138,277	13.00	6
Dibenz(ah)anthracene	278,139,276	13.03	6
Benzo(ghi)perlyene	276,138,277	13.36	6

5. An Initial calibration is considered acceptable, and the average response factor may be used for quantitation if the overall average of the %RSD is less than 25%. The continuing calibration verification is considered acceptable if the % difference from the curve is <= 20%. Pentachlorophenol and bis(2-ethylhexyl)phthalate will be allowed up to 40% difference in the continuing calibration verification.

6. To allow for the analysis of SIM and SCAN on the same extract, two additional surrogates are added to the samples prior to extraction. They are Fluorene-d10 and Pyrene-d10. They are added at a concentration of 1.0ng/ul in the final extract, and until sufficient data has been gathered to generate laboratory specific limits, an acceptance range of 30-150% recovery has been established for both waters and soils.

7. A SIM LCS will be extracted with every extraction batch (in addition to the SCAN LCS). The LCS will be spiked at 0.5ug/L (16.7ug/Kg for soils). Laboratory generated limits are used for all compounds. If there is insufficient data available to generate limits, an acceptance range of 30-150% will be used for both waters and soils. Re-extraction of the batch will not be required if bis(2-ethylhexyl)phthalate recovers above acceptance limits because as the spike amount approaches potential background levels, the apparent recoveries of this compound will be biased high.

8. If the client requests a SIM MS/MSD, it will be performed using the same spiking solution as is used for the LCS. The recovery limits will be the same as for the LCS, and the % RPD will be set at 50%. If the MS/MSD does not meet those limits, it will be noted in the case narrative.

9. The reporting limit will be set at the low point in the curve, which will be 0.1ug/L (3.3ug/Kg for soils) with the exception of pentachlorophenol, which will be 1.25ug/L (41.7ug/Kg for soils), and bis(2-ethylhexyl)phthalate, which will be 0.5ug/L(16.7ug/kg for soils). These limits can be decreased by a factor of 2 by concentrating the extracts to half the typical final volume.

10. Method blanks should not contain any target compound contamination greater than the reporting limit (with the exception of bis(2-ethylhexyl)phthalate, which may be 5 times the RL. However, since this is a trace analytical method there may be instances where this is not achieved. In that case, the client will be contacted to determine if re-extraction is required.

APPENDIX C

Simultaneous Sim/Scan Analysis

1. This appendix is to be used when simultaneous analysis is performed for both SIM and SCAN compounds. This analysis can only be performed using the Agilent 5975MS.

2. Prior to the start of any analysis, the instrument must meet the ion abundance criteria for 50ng or less of DFTPP injected in the scan mode. Additionally, the Electron Multiplier voltage in the DFTPP method must be identical to the voltage in the SIM method. Standards and/or samples can be analyzed for twelve hours from the injection of a compliant DFTPP Tune. The last injection must occur within a twelve hour period, but the run can end outside of the twelve hours.

3. The SIM portion of the method should contain a primary ion and at least one (but preferably two) confirmatory ions for each compound. There are some compounds for which only a primary ion is practical, such as Benzidine. The ions for the compounds will be placed in groups based on retention times of the compounds. The total cycle time for the ions in a group should not exceed one second. The dwell time for any ion in a group is typically 30-100msec.

4. The internal standard concentration shall be added at 5ul of a 100ppm solution, to result in a 10ng/ul on column concentration.

4. The initial calibration curve will consist of at least 5 points for each of the SIM and SCAN compounds. The typical calibration curve consists of the following 11 levels: 0.1ng/ul, 0.25ng/ul, 0.5ng/ul, 1.25ng/ul, 2.5ng/ul, 4.0/10ng/ul, 10/25ng/ul, 20/30ng/ul, 40ng/ul, 60ng/ul, and 80ng/ug. See Table 10 for a complete listing of individual compound concentrations for each level. The SIM compounds will typically have a working curve range of 0.1ng/ul – 80ng/ul. The Scan compounds will typically have a working curve range of 4ng/ul – 80ng/ul. This will vary for less sensitive compounds.

5. The continuing calibration shall have 2 analyses. The SIM continuing calibration level shall be the 0.5ng/ul. The SCAN analysis shall be at 50ng/ul.

6. The STL-CT SIM reporting list *with example retention times and suggested groupings) is presented below:

Compound	Ions	RT	Quant IS
Group 1			
Phenol	94,65,66		1
Aniline	93,66		1
2-Chlorophenol	128,64,130		1
1,4-dichlorobenzene-d4(I1)	152,115	3.02	n/a
Group 2			
2-Methylphenol	108,107		
4-Methylphenol	108,107		
Hexachloroethane	117,201,199		1
2,4-dichlorophenol	162,164,98		
Group 3			
Naphthalene-d8(I2)	136,68	4.25	n/a
Naphthalene	128,129,127	4.27	2
Hexachlorobutadiene	225,223,227	4.40	
2-Methylnaphthalene	142,141	4.97	2
Group 4			
Acenaphthylene	152,151,153	5.89	3
Acenapthene-d10(I3)	164,162,160	6.04	n/a
Acenapthene	153,154,1542	6.07	3
Group 5			
Fluorene-d10(S)	176,88,177	6.59	3
Fluorene	166,165,167	6.62	3
Hexachlorobenzene	284,142,249	7.17	
Group 6			

Pentachlorophenol	266,264,268	7.40	4
Group 7			
Phenathrene-d10(I4)	188,94,189	7.58	n/a
Phenathrene	178,179,176	7.61	4
Anthracene	178,176,179	7.66	4
Group 8			
Fluoranthene	202,101,100	8.85	4
Benzidine	184		
Pyrene-d10(S)	212,106,213	9.057	4
Pyrene	202,101,100	9.06	5
3,3'-Dimethylbenzidine	212,196		
Group 9			
3,3'-Dichlorobenzidine	252,254,126	10.33	
Bis(2-ethylhexyl)phthalate	149,167,279	10.41	5
Chrysene-d12(I5)	240,120,236	10.35	n/a
Benzo(a)anthracene	228,229,226	10.33	5
Chrysene	228,229,226	10.37	5
Group 10			
Benzo(b)fluoranthene	252,253,125	11.38	6
Benzo(k)fluoranthene	252,253,125	11.41	6
Benzo(a)pyrene	252,253,125	11.70	6
Perylene-d12(I6)	264,260,265	11.76	n/a
Group 11			
Indeno(1,2,3-cd)pyrene	276,138,277	13.00	6
Dibenz(ah)anthracene	278,139,276	13.03	6
Benzo(ghi)perlyene	276,138,277	13.36	6

7. An Initial calibration is considered acceptable, and the average response factor may be used for quantitation if the overall average of the %RSD is less than 25% for the SIM compounds. Linear regression or quadratic fit may be used as long as there are 5 levels available for linear regression and 6 for quadratic. The SCAN compounds must meet all normal requirements as stated in section 10.3 of this SOP. In instances where a SIM compound is listed as either a SPCC or CCC, that compound shall meet normal 8270 requirements of 0.05RRF or 30%RSD for the initial curve. Linear regression or quadratic fit may not be used on a compound listed as a CCC.

8. The continuing calibration verification for the SIM compounds is considered acceptable if the % difference from the curve is <= 20%. Bis(2-ethylhexyl)phthalate will be allowed up to 40% difference in the continuing calibration verification if it is required as a SIM compound. The continuing calibration verification for the SCAN compounds must meet the requirements stated in section 10.4 of this SOP.

9. To allow for the analysis of SIM and SCAN on the same extract, two additional surrogates are added to the samples prior to extraction. They are Fluorene-d10 and Pyrene-d10. They are added at a concentration of 1.0ng/ul or 2.0ng/ul (dependent on the final volume) in the final extract, and until sufficient data has been gathered to generate laboratory specific limits, an acceptance range of 30-150% recovery has been established for both waters and soils.

10. A SIM LCS and SCAN LCS will be extracted with every extraction batch. The LCS will be spiked at 0.5ug/L-1.0ng/uL (16.7-33ug/Kg for soils). Laboratory generated limits are used for all compounds. If there is insufficient data available to generate limits, an acceptance range of 30-150% will be used for both waters and soils. Re-extraction of the batch will not be required if bis(2-ethylhexyl)phthalate recovers above acceptance limits because as the spike amount approaches potential background levels, the apparent recoveries of this compound will be biased high. The SCAN LCS must meet the requirements outlined in section 11.5.2.

11. If the client requests a SIM MS/MSD, it will be performed using the same spiking solution as is used for the SIM-LCS. The

recovery limits will be the same as for the LCS, and the % RPD will be set at 50%. If the MS/MSD does not meet those limits, it will be noted in the case narrative.

12. The reporting limit will be set at the low point in the curve, which will be 0.1ug/L (3.3ug/Kg for soils) with the exception of pentachlorophenol, which will be 1.25ug/L (41.7ug/Kg for soils), and bis(2-ethylhexyl)phthalate, which will be 0.5ug/L(16.7ug/kg for soils). These limits can be decreased by a factor of 2 by concentrating the extracts to half the typical final volume. See Table 11 for a list of reporting limits typical for a 1000ml or 15g extraction with a 1ml final volume.

13. Method blanks should not contain any target compound contamination greater than the reporting limit (with the exception of bis(2-ethylhexyl)phthalate, which may be 5 times the RL. However, since this is a trace analytical method there may be instances where this is not achieved. In that case, the client will be contacted to determine if re-extraction is required.

Title: SOP for Sample - SPLP

[Method SW846 1312]

Approvals (Signature/Date):

Technical Manager Date 1/21/08

Health & Safety Manager / Coordinator Date 1/21/08

Quality Assurance Manager Date 1/21/08

Laboratory Director Date 1-21-08

This SOP was previously identified as SOP No. CVS06201.CT.

Any reference within this document to Severn Trent Laboratories, Inc. or STL, should be understood to refer to TestAmerica Laboratories, Inc. (formerly known as Severn Trent Laboratories, Inc.)

Facility Distribution No. _____ Distributed To:_____

1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 The SPLP is designed to determine the mobility of both organic and inorganic analytes present in solid, liquid and multi-phasic wastes.

2.2 If an analysis of any one of the liquid fractions of the SPLP extract indicates that a regulated compound is present at or above the regulatory level, the waste is considered to be hazardous.

2.3 The document control number for this SOP is CT-CVS-62, rev 2.

3.0 TERMS AND DEFINITIONS

3.1 Refer to the SOP for Laboratory Term and Definitions.

4.0 SUMMARY OF METHOD

4.1 For wastes containing less than 0.5 percent solids, the waste, after filtration through a 0.8 um glass fiber filter, is defined as the SPLP extract.

4.2 For wastes containing greater than 0.5 percent solids, the liquid phase, if any, is separated from the solid phase is reduced by cutting, crushing, or grinding (if necessary), weighed, and extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid used is a function of the alkalinity of the solid phase. A zero headspace extractor is used when volatile compounds are of concern.

4.3 If compatible, the initial phase of the waste is added to the liquid extract and analyzed following filtration. If incompatible, the liquids are analyzed separately and combined to yield a weighted average concentration.

4.4 This SOP is based upon EPA SW846 method 1312.

5.0 INTERFERENCES

5.1 Potential interferences that may be encountered during analysis are discussed in each of the individual analytical methods.

6.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, and this document.

6.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against organic solvents.

The rotary extraction device should be checked daily before use.

The use of a vacuum system during sample filtration presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced. Ensure that the vacuum exhaust hose is vented to a fume hood so vapors are not pumped into the working environment.

Pressure may build up in the extraction vessel. Vent into a hood if needed.

6.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure

Nitric Acid (1)	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydrator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

7.1 Samples should be stored unpreserved at 4°C, providing that refrigeration does not result in irreversible physical changes to the waste. Sample bottles are not reused.

7.2 When samples are to be analyzed for volatiles, care will be taken to minimize the potential loss of any volatiles.

7.3 The SPLP leaching for organic fractions must be performed within 14 days of sampling. Samples to be analyzed for mercury should be leached within 28 days, all other metals within 180 days.

8.0 APPARATUS AND MATERIALS

8.1 Top-loading balance, 0.01 g sensitivity

8.2 Agitation apparatus, 30 ± 2 rpm rotary mixer

8.3 Extraction vessels, glass or PTFE bottles with teflon-lined caps, 2L capacity

8.4 Orion 720 pH/ISE meter with combination pH electrode or equivalent

8.5 Drying oven, capable of maintaining 100 ± 20°C or equivalent

8.6 Desiccator

8.7 Glass fiber filters (0.8 um)

NOTE: Glass fiber filters require rinsing with 1.0 N HNO_3 followed by rinsing three times with reagent water.

8.8 Pressure filtration apparatus

8.9 Vacuum filtration appartus

8.10 Buchner funnels, 11 cm.

8.11 Side arm filter flasks, 1 & 2 L.

8.12 ZHE extractors.

8.13 Compressed Nitrogen Gas, ultra-high purity grade

8.14 Hot/stir plates

8.15 Beakers, 500, 150 mL

8.16 Spatula

8.17 Graduated cylinders, 1L, 100mL

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Laboratory prepared reagent (nanopure) water is used whenever reagent ASTM II Water (ASTM D11930) is required unless otherwise specified.

9.2 For volatiles, organic free reagent water stored in the volatiles lab is to be used.

9.3 Sulfuric acid/nitric acid (60/40 weight percent mixture).
Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid.

9.4 Extraction Fluid #1: This fluid is made by adding the 60/40 weight percent mixture of sulfuric acid and nitric acids to reagent water until the pH is 4.20 ± 0.05.

9.5 Extraction Fluid #2: This fluid is made by adding the 60/40 weight percent mixture of sulfuric acid and nitric acids to reagent water until the pH is 5.00 ± 0.05.

9.6 Extraction Fluid #3: This fluid is reagent water.

9.7 Analytical standards shall be prepared according to the appropriate analytical method.

10.0 CALIBRATION

10.1 Top-loading balance is calibrated daily to ± 0.01 g.

10.2 pH meter is calibrated prior to use with pH buffers 4, 7 and 10.

11.0 QUALITY CONTROL

11.1 All chemicals should conform to minimum specifications set by the Reagent Chemicals Committee of the American Chemical Society. All chemical inventories are used on a first-in first-out basis.

11.2 Employ a minimum of one blank per sample batch of 20 or fewer samples to determine if any contamination or any memory effects are occurring. Prepare a blank for each extraction fluid used.

11.3 Matrix spikes are required for each waste stream. The analytical groups will spike leachates prior to any preservation, after the leaching process.

11.4 Each agitation apparatus (spinner) is checked monthly to verify that the device rotates at 30 ± 2 rpms (the number of revolutions are counted for one full minute). The verifications are dated and recorded in the log book.

11.5 Record all NBS "S" weights where applicable.

12.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

12.1 Preliminary Evaluation: SPLP preliminary evaluations are performed on a minumum 100g aliquot of waste, this aliquot shall not actually undergo SPLP extraction.

12.1.1 Percent Solids Determination

If the waste will obviously yield no free liquid when subjected to vacuum or pressure filtration through a 0.8 um filter, proceed to step 12.1.2.

If the waste is liquid or multi-phasic, separate the phases. Weigh out a representative subsample of the waste (approximately 100 g). Allow slurries to stand, to permit the solid phase to settle, if applicable.

Weigh out a 0.8 um glass fiber filter. Transfer the waste to the filter holder (prepped with the tared filter) and apply vacuum, until all of the liquid has passed through the filter. Stop filtering when air passes through the filter. If this point is not reached, stop filtration when the liquid stops flowing.

Note: Oils which do not pass through the filter are treated as solids. Correct the initial sample mass for any material which adhered to the walls of the Millipore setup.

Remove the solid phase and filter media, while not allowing them to dry, weigh to ± 0.01 g. The wet weight of the residue is determined by the difference between the weight of the tared filter and the weight of the solid phase and filter.

Dry the filter and residue at 100°C ± 20°, and calculate the percent solids. The waste sample will be handled differently from this point depending on whether or not the waste contains more or less than 0.5 percent solids.

Note: This part of the procedure is used only to determine whether the solid must be extracted. Do not extract dried sample!

If the solid constitutes less than 0.5 percent of the waste, proceed to 12.2 for non-volatile parameters and 12.4 for volatiles.

12.1.2 Extraction Fluid Determination

For soils, if the sample is from a site that is east of the Mississippi River, extraction fluid #1 should be used. If the sample is from a site that is west of the Mississippi River, extraction fluid #2 should be used.

For wastes and wastewater, extraction fluid #1 should be used. For cyanide containing wastes and/or soils, extraction fluid #3 (reagent water) must be used.

12.1.3 Particle Size Determination

The solid phase of the waste must be crushed, cut, or ground in order that the solid may pass through a 9.5 mm or 0.375 in. standard sieve. If solids are being prepared for volatile fraction extraction, care must be taken to minimize exposure to the air, and prevent loss of volatiles.

12.2 Non-Volatile Fraction Extraction (no free liquid)

12.2.1 If the waste will obviously yield no liquid when subjected to pressure filtration, weigh out a 100g sample and place the waste into an extraction vessel.

12.2.2 Slowly add twenty times the mass of the waste of the appropriate extraction fluid to the extraction vessel. Close the extractor bottle tightly, secure in the rotary agitation device, and rotate at 30 rpm ± 2 rpm for 18 hours ± 2 hours. Ambient room temperature shall be maintained during the extraction period (23°C ± 2°).

12.2.3 At the end of the extraction period, the leachate is filtered through a 0.8 um glass fiber filter. Leachates are placed in amber glass bottles for semi-volatiles, herbicides and pesticides; polyethylene bottles are used for metals.

12.3 Non-Volatile Fraction Extraction (with free liquid)

12.3.1 If the sample is liquid or multi-phasic, perform the liquid/solid separation. The liquid filtrate will constitute part or all of the extract.

12.3.2 Pre-weigh the container that will receive the filtrate. Assemble the filter holder and filter. Acid-washed filters may be used for all non-volatile extractions.

12.3.3 Weigh out a subsample of the waste and record the weight. If the waste contains <0.5 percent dry solids, the liquid portion of the waste, after filtration, is defined as the SPLP extract. Enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required of the SPLP extract. For wastes containing >0.5 percent dry solids, enough solids must be generated by filtration to support the analyses to be performed on the SPLP extract.

12.3.4 Quantitatively transfer the waste aliquot to the filter holder, and filter. If waste material (>1 percent) of the waste has adhered to the filter apparatus top, determine the weight of the

residue and subtract it from the sample weight determined in Section 12.3.3 from the initial mass of the waste taken for extraction.

12.3.5 Quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase. Prepare the solid portion of the waste for extraction if required.

12.3.6 Determine the amount of extraction fluid to add to the extraction vessel (Section 12.3). Slowly add this amount of the appropriate extraction fluid. Close the extractor bottle tightly, secure in the rotary agitation device, and rotate at 30 rpm ± 2 rpm for 18 hours ± 2 hours. Ambient room temperature shall be maintained during the extraction period (23°C ± 2°).

12.3.7 Following the extraction period, the solution is filtered through 0.8 um glass fiber filters and placed unpreserved in bottles for the analytical groups.

Note: If any free liquid was collected prior to leaching, it should be combined with the leachate if compatible. If the two liquids are not compatible, they will be analyzed separately. The analytical results will be combined based on waste weight ratios following analysis.

12.3.8 Leachates are placed in amber glass bottles for semi-volatiles, herbicides and pesticides; polyethylene bottles are used for metals.

12.4 Volatile Extraction

12.4.1 Extracting Liquid Samples (<0.5% Solids)
Fill prepared, clean ZHE unit with liquid sample. Attach the nitrogen line, open the top valve, pressurize the unit expelling air from the unit. Close the top valve when all of the air has been expelled. Under pressure fill a minimum of three 40 mL volatile vials and label.

12.4.2 Extracting a Sludge Sample

Calculate the mass required to yield a 25 g dry weight sludge. Place the weighed waste aliquot into the ZHE unit and secure the lid. Attach the glass syringe to the ZHE unit using the Millipore interlock.

Attach the nitrogen line to the ZHE unit. Pressurize the unit to force the filterable liquid into the glass syringe.

Store the filtered liquid in 40 mL septa volatile vials at 4°C. If less than 40 mL of free liquid is available store the syringe containing the liquid at 4°C.

Attach nitrogen line to the unit containing the remaining non-filterable sludge; pressurize the unit to 1-10 psi.

Add extraction fluid to the ZHE vessel using a pressurized reservoir. When adding fluid: open the valve located on the base of the ZHE unit, to release the pressure and allow the ZHE to fill with fluid. Once fluid stops flowing into the ZHE: close the bottom valve and the line from the reservoir. Invert the unit three times to bring unwanted air bubbles to the top; release the top valve to expel air and eliminate the bubbles. Re-attach the reservior line and fill the remaining space left by expelling the air in the line. Pressurize the unit again to 1-10 psi.

Place the ZHE in the rotary agitation device and spin for 18 hours ± 2 hours. At the end of the extraction period draw off the leachate through an in-line glass fiber filter via a glass syringe. Place the leachate into volatile vials and refrigerate at 4°C until the time of analysis.

12.4.2 Extracting a Solid Sample

Place a 25 g aliquot into the ZHE unit and secure the lid. Add extraction fluid to the ZHE vessel using a pressurized reservoir. When adding fluid: open the valve located on the base of the ZHE unit, to release the pressure and allow the ZHE to fill with fluid. Once fluid stops flowing into the ZHE: close the bottom valve and the line from the pump. Invert the unit three times to bring unwanted air bubbles to the top; release the top valve to expel air and eliminate the bubbles. Re-attach the pump line and fill the remaining space left by expelling the air in the line. Pressurize the unit again to 1-10 psi.

Place the ZHE in the rotary agitation device and spin for 18 hours ± 2 hours. At the end of the extraction period draw off the leachate through an in-line glass fiber filter via a glass syringe. Place the leachate into volatile vials filling completely with no headspace and refrigerate at 4°C until the time of analysis.

13.0 CALCULATIONS

13.1 Percent Solids Calculation:

$$\text{Percent Solids} = \frac{\text{Weight of Solid Waste (g)} \times 100}{\text{Wet Sample (g)}}$$

13.2 Percent Dry Solids Calculation:

$$\%\ \text{Dry Solids} = \frac{(D-T) \times 100}{I}$$

where:

D = Weight of Dry Waste and Filter (g)
T = Tare weight of filter (g)
I = Initial weight of waste (g)

13.3 Amount of Extraction Fluid Calculation:

$$\text{Weight of Extraction Fluid} = \frac{20 \times \%\ \text{solids} \times \text{weight of waste}}{100}$$

13.4 ZHE Charge Calculation:

Weight of Waste to charge ZHE (g) = 2500/(% solids)

14.0 ACCEPTANCE OF DATA

14.1 Method blanks must not contain target analytes exceeding the practical quantitation limit (PQL) for that specified analyte. If a method blank fails surrogate recoveries or has a target analyte exceeding its PQL, then the QA manager is notified and a decision is made on how to correct the situation.

15.0 REPORTING OF RESULTS

15.1 Not Applicable.

16.0 POLLUTION PREVENTION

16.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a

variety of techniques. These include the following.

16.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

16.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

16.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

16.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

16.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

17.0 WASTE MANAGEMENT

17.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

17.2 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Acidic aqueous sample waste. Employees are to collect and dispose of this material in satellite waste accumulation drums..
- Solid sample waste and used filter paper from the sample filtration step. Employees are to collect and dispose of this material in satellite waste accumulation drums.
- Glassware contaminated with acidic sample residue. Employees are to collect and dispose of this material in satellite waste accumulation drums.

18.0 SUPPLEMENTAL DOCUMENTS

18.1 SOP for Laboratory Term and Definitions.

19.0 REFERENCES

19.1 Test Methods for the Evaluation of Solid Waste, SW846, 3rd Edition, Method 1312.

20.0 SUBSTANTIVE REVISIONS

20.1 Original document.
20.2 Revisions – 01/27/06
- i) Added Health and Safety officer signature to section 1.0.
- ii) Updated safety information in section 6.0.
- iii) Updated waste management information in section 17.0.

20.3 Added new TestAmerica SOP header and control number, changed name, 01/21/08.

Title: Prep of Soil Samples for Organochlorine Pestcides/PCBs
[Method(s) SW846 3550B]

Approvals (Signature/Date):

Technical Manager Date 12/05/07

Health & Safety Manager / Coordinator Date 12-5-07

Quality Assurance Manager Date 11/30/07

Laboratory Director Date 12-5-07

This SOP was previously identified as SOP No. SPS01608.CT.

Facility Distribution No. _____ **Distributed To:_____**

1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 This method is used to extract organochlorine pesticides and Aroclors from soil/sediment samples.

2.2 It is the policy of TestAmerica and of the Organic Extractions Group to ensure that we administer contracts and orders for goods and services in a manner that is fully compliant with governmental laws and regulations, as well as the TestAmerica Policy Statement on Business Ethics and Conduct.

2.3 The document control number for this SOP is CT-SPS-16, Rev 8.

3.0 TERMS AND DEFINITIONS

3.1 There are many definitions used with in the laboratory, which may be generic to all laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used with in the laboratory, reference the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 Soil/sediment samples are serially extracted using an ultrasonic probe with 1:1 acetone/methylene chloride or acetone/hexane. The extract is concentrated using a Kuderna-Danish (K-D) apparatus. If higher molecular weight compounds that interfere with the analysis are present, GPC cleanup can be performed.

4.2 This SOP is based on EPA SW846 Method 3550B - Sonication Extraction. The concentration of the matrix spike compounds used in these procedures differs from those recommended in Method 3500. To eliminate the dilution caused by GPC cleanup the final volume after GPC cleanup has been modified. The analyst is referred to these methods for further information about the procedures.

5.0 INTERFERENCES

5.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and

other sample processing apparatus. This can lead to discrete artifacts and/or elevated baselines in the gas chromatograph. All these materials must be demonstrated to be free from interferences by the running of reagent (method) blanks. A specific interference that can cause a problem is the presence of phthalate esters, which are commonly found in plastics. Those interferences can be avoided by not using plastics in the laboratory.

5.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source.

6.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

6.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard. The KD apparatus has ground glass joints which can become stuck. Technicians must use Kevlar or other cut/puncture resistant gloves when separating stuck joints.

6.2 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure

Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.3 Material Safety Data Sheets for all chemicals used in the operation are present in the laboratory for immediate access.

7.0 SAMPLE PRESERVATION AND STORAGE

7.1 Soil/sediment samples must be protected from light and refrigerated at 4°C from the time of receipt until extraction and analysis as detailed in "Storing Water and Soil Samples for Organic and Inorganic Sample Analysis".

7.2 Soil samples shall be extracted within 14 days of collection. Extracts must be analyzed within 40 days of extraction.

7.3 After concentration, samples will be placed in a designated refrigerator in the GC lab. The extract Chain of Custody must be completed (Figure 1).

8.0 APPARATUS AND MATERIALS

8.1 Glass beakers, 400 mL.

8.2 Side arm flask, 500 mL.

8.3 Concentrator tube, K-D, 10 ml, graduated. The calibration must be checked at the volumes employed in the test using balance. If the calibration is not accurate, do not put the tube into service. Return the tube to the manufacturer for a credit. Document the results in the Concentrator Tube Calibration Logbook according to the procedures detailed below for

every concentrator tube received.

8.3.1 Record the temperature of the water being used to calibrate the receivers at the top of the page.

8.3.2 Place the tube to be calibrated in a container on a 4 place analytical balance, and zero the balance.

8.3.3 Add reagent water to the 0.5 ml mark, and reweigh. The weight must be 0.5g$\pm$ 0.015g.

8.3.4 Add reagent water to the 1.0 ml mark, and reweigh. The weight must be 1.0g $\pm$ 0.03g.

8.3.5 Add reagent water to the 5 ml mark, and reweigh. The weight must be 5.0g. $\pm$ 0.15g.

8.3.6 Add reagent water to the 10 ml mark, and reweigh. The weight must be 10.0g. $\pm$ 0.3g.

8.3.7 If the tube does not meet criteria at any of these points, do not put it in service.

8.4 Evaporative flask, K-D, 500 mL.

8.5 Snyder column, 3-ball macro.

8.6 Vials, clear, 7 mL and 4mL capacity with teflon lined screw cap.

8.7 Buchner funnels, 100 mm diameter.

8.8 Disposable pasteur pipets, cotton plugged.

8.9 Pyrex glass wool, rinsed with methylene chloride.

8.10 Silicon carbide boiling chips, approximately 10/40 mesh, solvent extracted.

8.11 Water bath, heated, capable of temperature control ($\pm$ 5°C), should be in a hood.

8.12 Balance, analytical, capable of weighing ±0.01 g.

8.13 Nitrogen evaporation device equipped with a heated water bath that can be maintained at 35-40°C.

8.14 Sonicator, equipped with 3/4" disrupter horn and a minimum of 375 watt capability.

8.14.1 Make sure the proper probes are connected for tuning as per manufacturers specifications. Only a factor on Unit #1 & #2 – only lower probe should be connected.

Turn the sonicator on, if not already.

Flip the selector switch to tune or press tune button.

Unit #1& #2 (TM-600-2) – The tune switch on the left, immediately to the right of the power switch. The tune adjustment knob is just to the right of the tune switch.
Unit #3&#4(XL2020) – The "tune" button is on the keypad. The tune adjustment knob is on the lower front right side of the box.

When performing the tune, the probes must not be in solvent, and the power should not be applied for longer than 10 second intervals. Adjust reading to lowest level possible.

Switch unit back to operational mode.

8.15 Syringes, various sizes, gas-tight.

8.16 Oven, drying, capable of maintaining 135°C (±2°C).

8.17 Desiccator.

8.18 Weighing dishes.

8.19 Whatman #41 filter paper, 100 mm (or equivalent).

8.20 GPC, Accuprep 3300. See the SOP for "GPC of Pesticide/PCB Extracts - Method 3640" for information concerning GPC cleanup.

8.21 Balance, top loading, capable of weighing ±0.01 g.

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Acetone, Methylene Chloride, Methanol, Hexane - Pesticide quality or equivalent.

9.2 Sodium sulfate - (ACS) granular anhydrous, purify by placing in muffle furnace at 400°C for a minimum of 4 hours.

9.3 Surrogate working solution - Prepare a solution of TCX/DCB at a concentration of 0.2 ug/mL in acetone. The solution must be stored at 4°C (±2°C) in amber teflon sealed containers for a maximum of 6 months. Source: Supelco

9.4 Pesticide matrix spiking solution - The spiking solution shall contain the following pesticides at the specified concentrations in acetone:

Pesticide	Concentration, ug/mL
Lindane	0.5
Heptachlor	0.5
Aldrin	0.5
Dieldrin	1.0
Endrin	1.0
4,4'-DDT	1.0

The solution must be stored at 4°C (±2°C) in teflon sealed amber containers for a maximum of 6 months. Source: Supelco.

9.4.1 PCB Matrix Spike Solution - If a project is also for PCB analysis, an Aroclor matrix spike will be performed using Aroclor 1260 at a concentration of 2 ug/ml. Source: Supelco.

9.5 Pesticide QC Check Solution (LCS Solution) - The spiking solution shall contain the compounds listed in Table 1 in methanol or acetone. The solution must be stored at room temperature in teflon sealed amber containers for a maximum of 2 weeks. All compounds are present at a concentration of 0.2 ug/mL. Source: Supelco.

9.5.1 PCB QC Check Solution (LCS Solution) - this solution is used for samples requiring PCB analysis also. The solution consists of Aroclor 1242 and Aroclor 1260 at a concentration of 10 ug/ml. The solution must be stored at 4°C (±2°C) in a teflon sealed amber container for a maximum of 6 months. Source: Supelco.

10.0 CALIBRATION - N/A

11.0 QUALITY CONTROL

11.1 A minimum of one blank (consisting of 30 g of sodium sulfate spiked with surrogate and carried through the entire analytical procedure) will be extracted every day samples are processed. A maximum of 20 samples can be extracted under a method blank in a calendar day.

11.2 A matrix spike/matrix spike duplicate will be extracted at a frequency of 1 per case or 20 field samples in an SDG. However, if more than 7 days elapses and 20 samples have not been extracted, it will be necessary to extract an MS/MSD.

11.3 All logbooks and forms will be dated and filled out with black ink, and any errors must be crossed out with a single black line. The change must be signed and dated by the person making the change. In no case will the original entry be made unreadable. Any unused portion of the logbook page will be "Z"ed out. The logbook will be reviewed and approved

periodically by the Group Leader or their designee.

11.4 Any bottled solvents used must come from lots of solvent approved by the corporate QA Officer using the procedures established in the "Corporate SOP for Solvent Approval". Solvents used from cycletainers must be received with a certification of analysis from the manufacturer before use.

11.5 Any problems encountered in the course of analysis (i.e. extracts going dry, extract spillage, improper pH, etc.) shall be documented by noting in the case narrative and by filling out a Corrective Action Report and/or NCM. The filled out CAR will be forwarded to the Group Leader or his/her designee who will give it to the QA Officer. The QA Officer will give the CAR to the Project Manager who will contact the client for instructions on how to proceed.

11.6 All glassware used in processing samples must be cleaned in accordance with the "SOP for Cleaning Glassware".

11.7 The extractions Batch Approval Sheets must be dated and filled out on a daily basis. They are located on the back of the extractions logbook page (Figure 3). There is also space provided for extraction information to be included in the case narrative.

11.8 The Extractions Logbook must be completely filled out on a daily basis for every batch extracted or concentrated (Figure 4). The logbook will be reviewed and approved at periodically by the Group Leader or his/her designee.

11.9 The lot numbers of reagents used in the extraction and concentration must be recorded in the reagents section of the Extractions Logbook Page.

11.10 The Extractions refrigerator and balance will be checked daily using the "SOP for Temperature Monitoring of Ovens, Refrigerators and Freezers".

11.11 All analysts will perform an initial demonstration of extraction proficiency. The documentation will be maintained by the QA Officer.

11.12 An LCS will be extracted at the same frequency as the method blank.

12.0 SAMPLE PREPARATION

12.1 Chain of Custody Procedures

12.1.1 All analysts are responsible for maintaining sample custody in accordance with EPA guidelines as detailed in "Documenting Sample Removal from the Laboratory".

12.1.2 All samples must be signed out on the chain of custody forms located in sample control.

12.2 Sample Extraction/Concentration - Low Level

12.2.1 When the sample is brought to the laboratory from sample control, the extraction logbook for the batch is started. Figure 4 is an example of an Extraction Logbook Page. The figure is broken down into blocks that the analyst will fill out on the actual logbook page at various parts of the extraction process as detailed in this SOP. Each section of the figure has a letter in it designating who has responsibility for completing the section. The person extracting is designated by an "E", and the person doing the concentration is designated by a "C". Enter the appropriate sample and solvent lot information on the logbook page.

a) If multiphase samples are received, fill out a CAR and do not extract the sample until the client has been informed and provides the lab with instructions on how to proceed.

12.2.2 If there is free water present in the sample, decant and discard the excess water on the sample. Record the presence of free water in the comments section of the extractions logbook.

12.2.3 With every batch of samples processed, a blank must be extracted. The blank will consist of 30 g of sodium sulfate spiked with surrogate and carried through the entire analytical procedure.

12.2.4 To ensure that every sample is properly spiked, the process must be witnessed by another analyst. The witnessing analyst will also initial the extractions logbook.

12.2.5 Weigh 30.0 g ±0.9 g into a labeled 400 mL beaker.

a) If an MS/MSD is to be run with the batch, prepare two additional aliquots of the sample as above. The frequency of MS/MSD analysis is specified in Section 10.2.

12.2.6 Add 1.0 mL of pesticide surrogate to every sample, blank, sample and spike/matrix spike duplicate (MS/MSD) and QC check sample (LCS) with a 1 mL syringe. If an MS/MSD is to be run with a batch, add 1.0 mL of the appropriate matrix spike solution to the MS/MSD samples with a 1 mL syringe.

a) For NYSDEC SW846, any time an MS/MSD is extracted it is also necessary to extract an MSB (matrix spike blank). Add 1000 uL of the appropriate matrix spike to a purified sodium sulfate blank. Call it "BlankID MSB" (e.g. 112200-B01MSB).

12.2.7 Pesticides - for the QC check sample (LCS) to be run with every batch, add 100 uL of the pesticide QC check solution to purified sodium sulfate blank. Call it "BlankID QC" (e.g. 112200-B01QC).

PCBs - For the QC check sample to be extracted with every batch, add 1 ml of PCB QC

check solution to the purified Na2SO4 blank.

12.2.8 Add approximately 60g of anhydrous sodium sulfate and mix well. Samples should have a sandy texture. Additional sodium sulfate may be added if necessary to achieve sandy texture. Immediately add 100 mLs of 1:1 acetone/hexane or methylene chloride/acetone to the sample. The appropriate information is entered into the surrogate and spike blocks of the extraction logbook.

12.2.10 Sonicate the samples for 3 minutes using the 3/4" horn with the output control knob set at 10 and a pulse rate of 50 percent. This is equivalent to a 1 second pulse. The bottom of the sonicator horn should be about 1/2" below the surface of the solvent, but above the sediment layer.

a) It may be necessary to break up lumps in the sodium sulfate using a clean spatula prior to extraction.

12.2.11 Decant the extract and filter through a Whatman #41 filter paper pre-wet with the 1:1 acetone/hexane or acetone/methylene chloride using vacuum filtration into a labeled side arm flask.

a) The label should include the sample number and the parameter analyzed. Colored tape is used for the label.

12.2.12 The extraction is repeated twice with 2 additional 100 mL portions of 1:1 acetone/hexane (or acetone/methylene chloride). Decant off the solvent after each extraction as in Section 12.2.10.

12.2.13 After the final sonication, the entire sample is poured into the Buchner funnel and rinsed with approximately 40 mLs 1:1 acetone/hexane or acetone/methylene chloride. Initial the appropriate extraction and spiking blocks in the extractions logbook.

12.2.14 Transfer the extract to a K-D concentrator. Rinse the side arm flask with 10-30 mLs of hexane to ensure all of the extract is transferred to the K-D flask. Transfer the tape label to the K-D flask.

12.2.15 Add one or two clean boiling chips to the K-D and attach a 3-ball snyder column. Pre-wet the snyder column with about 1 mL methylene chloride. Place the K-D in a hot water bath (80-85□C) so that the concentrator tube is partially immersed in the hot water bath and the entire lower rounded surface of the flask is bathed with hot vapor. The temperature should be such that the concentration is completed within 20-25 minutes. When the apparent volume reaches 3-4 mLs, remove the K-D and add 60 mLs of hexane to the K-D through the snyder column, and concentrate as above. When the apparent volume reaches 3-4 mLs, remove from the batch and allow the K-D to cool.

a) At the proper rate of distillation, the balls in the column will actively chatter but the chambers will not flood with condensed solvent.

b) If GPC cleanup is required, remove the snyder column, rinse the flask and its lower joint into the receiving vessel and adjust the volume to 10 mLs with methylene chloride. Proceed with the GPC cleanup according to the latest version of the SOP for "GPC Cleanup of Pesticide Extracts Method 3640". After GPC cleanup, concentrate as above and proceed with the solvent exchange to hexane.

12.2.16 Remove the snyder column, and rinse the flask and its lower joint into the concentrator tube with hexane. Bring the extract to a final volume of 10 mLs. It may be necessary to blow down the extract by placing the concentrator tube in the N-Evap (maintained at 30-35°C) and evaporating the solvent using a gentle stream of clean, dry nitrogen. During the concentration step, the solvent level must be kept below the water level of the batch. The extract must never be allowed to go dry.

12.2.17 If sample is PEST only, transfer the extract to a labeled 7mL vial. If sample is PCB only or PEST/PCB, transfer a portion of the extract to a labeled 7mL vial and a portion into a 4mL vial(used for acid C/U). Initial the concentration block of the extractions logbook.

a. For PCB samples, take the extract that is in the 4mL vial and add approximately 1-1.5mL of sulfuric acid using a Pasteur pipet. Cap the extract, shake 2 times and then vent. Tighten the cap and shake for 10-15 seconds. Let the sample settle for at least 30 seconds. Using a pipet, transfer the cleaned sample extract(the top layer) into a clean labeled 4mL vial. If the sample extract is still dark in color, additional acid clean-ups may be done. Follow the same steps as above.

12.2.18 The PCB/pesticide extract is now ready for analysis. Place the extract in the designated refrigerator in the GC lab maintained at 4°C and complete the Extract Chain of Custody (Figure 1).

12.3 If GPC cleanup is required, the sample must be concentrated to 10 mls in methylene chloride. For information on performing GPC cleanup, see the latest version of the SOP for "GPC of Pesticide/Aroclor Extracts - Method 3640".

12.3.1 After GPC cleanup, transfer the "cleaned up" extract to a K-D concentrator. Rinse the flask with 20-30 mL of methylene chloride to quantitatively transfer the sample. Transfer the tape label to the K-D flask.

12.3.2 Add one or two clean boiling chips to the K-D and attach a 3-ball snyder column. Pre-wet the snyder column with about 1 mL methylene chloride. Place the K-D in a hot water bath (80+/-5□C) so that the concentrator tube is partially immersed in the hot water bath and the entire lower rounded surface of the flask is bathed with hot vapor. The temperature should be such that the concentration is completed within 20-25 minutes. When the apparent

volume reaches 3-4 mLs, remove the K-D and add 60 mLs of hexane to the K-D through the snyder column, and concentrate as above. When the apparent volume reaches 3-4 mLs, remove from the batch and allow the K-D to cool.

a) At the proper rate of distillation, the balls in the column will actively chatter but the chambers will not flood with condensed solvent.

12.3.3 Remove the snyder column, and rinse the flask and its lower joint into the concentrator tube with hexane. Bring the extract to a final volume of 5 mLs by placing the concentrator tube in the N-Evap (maintained at 30-35°C) and evaporating the solvent using a gentle stream of clean, dry nitrogen. During the concentration step, the solvent level must be kept below the water level of the batch. The extract must never be allowed to go dry.

12.3.4 Transfer the extract to a labeled vial. Initial the concentration block of the extractions logbook.

12.3.5 The PCB/pesticide extract is now ready for analysis. Place the extract in the designated refrigerator in the GC lab maintained at 4°C and complete the Extract Chain of Custody (Figure 1).

12.4 Sample Preparation - Medium Level - for samples expected to contain high concentrations of Organics (greater than 20 mg/Kg).

12.4.1 If there is any water layer on the sample, decant and discard, and thoroughly mix the sample and discard any foreign objects (eg. sticks, rocks, etc.). In a fume hood weigh 2.0 ±0.1 g sample into a 22 mL vial. Wipe the mouth of the vial to remove any excess material and record the exact weight in the extractions logbook. Cap the vial to avoid cross contamination.

a) If an MS/MSD is to be run with the batch, weigh out 2 additional 2 g aliquots. The frequency of MS/MSD analysis is specified in Section 10.2.

12.4.4 Weigh out 2.0 g sodium sulfate as a blank for each batch of samples. The blank is spiked with surrogates and carried through the entire analytical procedure.

12.4.5 Add 2.0 g of anhydrous sodium sulfate to the sample and blanks and mix well.

12.4.6 Add 1 ml of surrogate to all samples, blanks, MS/MSD, and the QC check using a syringe.

12.4.7 If an MS/MSD is to be run with a batch of samples, add 1 ml of the appropriate matrix spiking solution to the MS/MSD samples using a syringe.

a) For NYSDEC SW846, any time an MS/MSD is extracted it is also necessary to extract an MSB (matrix spike blank). Add 1 ml of the appropriate matrix spike solution

to a purified sodium sulfate blank spiked with surrogates. Call it "BlankID MSB" (e.g. 112200-B01MSB).

12.4.8 For the QC Check to be run with every batch, add 1 ml of each of the appropriate QC check solution to a purified sodium sulfate blank spiked with surrogates. Call it "Blank IDQC" (eg. 112200-B01QC).

12.4.8 Immediately bring up to 10 mLs with Hexane. Sonicate the sample for 2 minutes using the 1/8" tapered microtip horn. The output control knob is set at 5 and with the mode switch on pulse and percent duty cycle at 50%.

a) Prior to starting the extraction, make sure that the sodium sulfate is free flowing. It may be necessary to break up the lumps with a spatula.

12.4.9 Loosely pack disposable Pasteur pipets with 2-3 cm glass wool plugs. Filter the extract through the glass wool and collect the extract in a clean sample vial. (The entire 10 mls of solvent cannot be recovered from the sample).

12.4.10 The extract is now ready for analysis. Place the extracts in the designated refrigerator maintained at 4°C and fill out the extract chain of custody located in the instrument lab (Figure 1). The extract must be analyzed within 40 days from extraction.

13.0 CALCULATIONS - N/A

14.0 ACCEPTANCE OF DATA - N/A

15.0 REPORTING OF RESULTS

15.1 Copies of logbook pages, any CAR's, and GPC chromatograms will be put in the client job folder.

16.0 SUPPLEMENTAL DOCUMENTS

16.1 "Storing Water and Soil Samples for Organic and Inorganic Sample Analysis".

16.2 "GPC of Pesticide/Aroclor Extracts - Method 3640A".

16.3 "SOP for SW846 Extractions Standards Preparation".

16.4 "SOP for Solvent Assays".

16.5 "SOP for Cleaning Glassware".

16.6 "Documenting Sample Removal from the Laboratory".

16.7 "SOP for Temperature Maintenance of Ovens, Refrigerators and Freezers".

17.0 POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

17.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

17.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

17.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

17.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

17.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

17.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

18.0 WASTE MANAGEMENT

18.1 All waste shall be managed in accordance with all state and federal requirements. See the STL-CT RCRA Contingency Plan.

18.2 Personnel who handle or generate waste must be trained within six months of employment in proper waste handling and requirements.

18.3 Waste Streams Produced by the Method
The following waste streams are produced when this method is carried out.

- Used samples, sodium sulfate, and glass wool or filter paper contaminated with

methylene chloride from the extract drying step. This waste is collected in day containers labeled for non-regulated solid waste. This waste is then transferred daily to the large waste drum labeled for non-regulated solid waste in the Waste Storage room.

- Assorted flammable solvent waste from various rinses. This waste is collected in a 2 liter beaker labeled for mixed waste. When this is full it is poured in a day container labeled for mixed waste. This is transferred daily to a large waste drum labeled for mixed waste in the Waste Storage room.
- Methylene chloride waste from various rinses. Methylene chloride waste is collected in a labeled 2 liter beaker then disposed of in the waste drum labeled for methylene chloride waste in the in the Waste Storage room.

Miscellaneous disposable glassware contaminated with acids, caustics, solvents and sample residue.

19.0 REFERENCES

19.1 USEPA SW846 - Test Methods for the Evaluation of Solid Waste, 3rd Ed.

20.0 SUBSTANTIVE REVISIONS

20.1 Original issue - 01/21/94.

20.2 Changed IEA to AEN. Changed extraction solvent from acetone/methylene chloride to acetone/hexane for samples that do not require GPC cleanup. Removed SOP code references; 10/15/97.

20.3 Changed AEN to STL, Updated method to reflect correct revision letter; 8/28/98.

20.4 Revised section 7.3 to replace the receiver calibration procedure, Added sections 9.4.1 and 9.5.1 for PCB standards, revised sections 11.2.8 and 11.2.9 to include PCB spiking, Added section 12.4 to address Medium Level extraction procedure; 03/05/99.

20.5 Added Terms and Definitions section, Pollution Prevention, and Waste Management; 2/15/2000. Also revised the section on Summary of Method, took out the deviation of using decachlorobiphenyl instead of dibutyl chlorendate. The most recent update specifies decaclorobiphenyl now.

20.6 Minor revisions to reflect current practices and added statement regarding water temperature to the receiving vessel calibration requirements - 6/20/02 (HR).

20.7 Added section 8.14.1, sonicator tuning procedure, 4/20/02 MKC.

20.8 Minor Revisions to reflect current practices and added a statement regarding current safety and waste management/ pollution prevention requirements – 5/6/04 (WDG).

20.9 Changed STL to TestAmerica. Added acid clean-up procedure for PCB extracts. Minor revisions to reflect current practices. 07/18/07 sbw

20.10 Added source of surrogates and spikes. Took out "addition of acid to water…" comment in section 6.2 as it is not applicable to soil extraction. Added new TestAmerica SOP header and control number, updated waste disposal; 10/26/07 sbw

TABLE 1.0
PESTICIDE QC CHECK (LCS) AND FULL MATRIX SPIKE, UG/ML

Analyte	Stock	Standard for Extractions
Aldrin	1.0	0.20
Dieldrin	1.0	0.20
p,p'DDT	5.0	1.0
p,p'DDE	1.0	0.2
p,p'DDD	5.0	1.0
Endosulfan I	1.0	0.20
Endosulfan II	5.0	1.0
Endosulfan Sulfate	5.0	1.0
Endrin	5.0	1.0
Endrin Aldehyde	5.0	1.0
Heptachlor	1.0	0.20
Heptachlor Epoxide	1.0	0.20
alpha-BHC	1.0	0.20
beta-BHC	1.0	0.20
gamma-BHC	1.0	0.20
delta-BHC	1.0	0.20
Methoxychlor	10.0	2.0
Endrin Ketone	5.0	1.0
4,4'DDT	1.0	0.20

Title: Prep of Water Pesticide/PCB Extracts

[Method(s SW846 3510C/3520C)]

Approvals (Signature/Date):

Technical Manager Date 12/05/07

Health & Safety Manager / Coordinator Date 12-5-07

Quality Assurance Manager Date 11/30/07

Laboratory Director Date 12-5-07

This SOP was previously identified as SOP No. SPS01207.CT.

Any reference within this document to Severn Trent Laboratories, Inc. or STL, should be understood to refer to TestAmerica Laboratories, Inc. (formerly known as Severn Trent Laboratories, Inc.)

Facility Distribution No. _____ Distributed To:_____

1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 This method is used to extract pesticides and Aroclors from water samples.

2.2 It is the policy of TestAmerica and of the Organic Extractions Group to ensure that we administer contracts and orders for goods and services in a manner that is fully compliant with governmental laws and regulations, as well as the TestAmerica Policy Statement on Business Ethics and Conduct.

2.3 The document control number for this SOP is CT-SPS-12, Rev 7.

3.0 TERMS AND DEFINITIONS

3.1 There are many terms and definitions used within the laboratory, which are listed in the latest version of the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 Aqueous samples are either serially extracted in a separatory funnel or extracted for 18 hours in a continuous extractor using methylene chloride. The extract is dried using sodium sulfate, and concentrated using a Kuderna-Danish (K-D) apparatus. If higher molecular weight compounds that interfere with the analysis of target compounds are present, GPC cleanup can be performed. The extract is exchanged to hexane, and is then ready for analysis.

4.2 This SOP is based on EPA SW846 Methods 3510C - Separatory Funnel Extraction and 3520C - Continuing Liquid/Liquid Extraction. The analyst is referred to these methods for further information about these procedures. The concentration of the acid surrogate and matrix spike compounds used in these procedures differs from those recommended in Method 3500. Also, DCB has been substituted for DBC in the surrogate solution and the concentration of the surrogate solution differs from that recommended in Method 3500. To eliminate the dilution caused by GPC cleanup, the surrogate/spike amount added and the final volume after GPC cleanup have been modified.

5.0 INTERFERENCES

5.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus. This can lead to discrete artifacts and/or elevated baselines in the gas chromatograph. All these materials must be demonstrated to be free from interferences by the running of reagent (method) blanks. A specific interference that can cause a problem is the presence of phthalate esters, which are commonly found in plastics. Those interferences can be avoided by using clean solvents and not permitting samples to contact plastics in the laboratory.

5.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source.

6.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document.

6.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard. The KD apparatus and glass separatory funnels have ground glass joints which can become stuck. Technicians must use Kevlar or other cut/puncture resistant gloves when separating stuck joints.

The use of separatory funnels to extract aqueous samples with Methylene Chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted, periodic venting may be necessary during the extraction. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, the use of a face shield over safety glasses or goggles is recommended. Keep the sash on the fume hood as low as reasonably possible.

6.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method.** The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydrator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 SAMPLE PRESERVATION AND STORAGE

7.1 Water samples must be collected in amber bottles and protected from light and refrigerated at 4□C from the time of receipt until extraction and analysis as detailed in "Storing Water and Soil Samples for Organic and Inorganic Sample Analysis".

7.2 Water samples shall be extracted within 7 days of collection. Samples extracted using NYSDEC SW846 protocol samples shall be extracted within 5 days VTSR (Validated Time of Sample Receipt), and concentrated within 7 days VTSR.

7.3 After concentration, samples will be placed in a designated refrigerator in the GC lab. The analyst will fill out the extract chain of custody in the extract chain of custody logbook located next to the refrigerator (Figure 1).

8.0 APPARATUS AND MATERIALS

8.1 Separatory funnel, 2,000 mL with Teflon stopcock.

8.2 Powder Funnel

8.3 Concentrator tube, K-D, 10 ml, graduated. The calibration must be checked at the volumes employed in the test using balance. If the calibration is not accurate, do not put the tube into service. Return the tube to the manufacturer for a credit. Document the results in the Concentrator Tube Calibration Logbook according to the procedures detailed below for every concentrator tube received.

8.3.1 Record the temperature of the water being used to calibrate the receivers at the top of the page.

8.3.2 Place the tube to be calibrated in a container on a 4 place analytical balance, and zero the balance.

8.3.3 Add reagent water to the 0.5 ml mark, and reweigh. The weight must be 0.5g$\pm$ 0.015g.

8.3.4 Add reagent water to the 1.0 ml mark, and reweigh. The weight must be 1.0g $\pm$ 0.03g.

8.3.5 Add reagent water to the 5 ml mark, and reweigh. The weight must be 5.0g. $\pm$ 0.15g.

8.3.6 Add reagent water to the 10 ml mark, and reweigh. The weight must be 10.0g. $\pm$ 0.3g.

8.3.7 If the tube does not meet criteria at any of these points, do not put it in service.

8.4 Evaporative flask, K-D, 500 mL.

8.5 Snyder column, 3-ball macro.

8.6 Vials, amber, 2-20 mL capacity with Teflon lined screw cap.

8.7 Continuous liquid/liquid extractor setup with glass connecting joints and/or Teflon crossover tube.

8.8 Erlenmeyer flask, 250 or 500 mL, or 500mL amber jar.

8.9 Graduated cylinder, 1,000 mL.

8.10 Pyrex glass wool, rinsed with methylene chloride before use.

8.11 Silicon carbide boiling chips, approximately 10/40 mesh, heated in the muffle furnace at 400□C for a minimum of 5 hours.

8.12 Water bath, maintained at 80-90□C, with rings, should be in a hood.

8.13 Balance, analytical, capable of weighing ±0.0001 g.

8.14 Nitrogen evaporation device equipped with a water bath that can be maintained at 35-40□C.

8.15 Chlorine test kit or chlorine tabs, capable of detecting 0.1 ppm residual chlorine.

8.16 Syringes, various sizes, gas-tight.

8.17 GPC, ABC Labs Model 1002B or Model AP1000.

8.18 Filter paper

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Reagent water - Water in which a contaminant is not observed at or above one half the PQL for each analytical parameter when one liter is extracted.

9.2 Acetone, hexane, methylene chloride - Pesticide quality or equivalent.

9.3 Sodium sulfate - (ACS) granular anhydrous, purify by placing in muffle furnace at 400□C for a minimum of 4 hours.

9.4 Sodium hydroxide solution, (10N) - Dissolve 400 g NaOH in reagent water and dilute to 1000 mL.

9.5 Sulfuric acid - (ACS)

9.6 Surrogate working solution - Prepare a solution of tetrachloro-m-xylene and decachlorobiphenyl at a concentration of 0.2 ug/mL each in acetone. The solution must be stored at 4□C (±2□C) in Teflon sealed amber containers for a maximum of 6 months. Source: Supelco.

9.7 Pesticide matrix spiking solution - The spiking solution shall contain the following pesticides at the specified concentrations in methanol:

Pesticide	Concentration, ug/mL
Lindane	0.5
Heptachlor	0.5
Aldrin	0.5
Dieldrin	1.0
Endrin	1.0
4,4'DDT	1.0

The solution must be stored at 4□C (±2□C) in Teflon sealed amber containers for a maximum of 6 months. Source: Supelco.

9.7.1 PCB Matrix Spike Solution - If a project is also for PCB analysis, an Aroclor matrix spike shall be performed using Aroclor 1260 at a concentration of 2 ug/ml. Source: Supelco.

9.8 Pesticide QC Check Solution (LCS Solution) - The spiking solution shall contain the compounds listed in Table 1 in methanol or acetone. The solution must be stored at 4□C (±2□C) in Teflon sealed containers for a maximum of 2 weeks. Source: Supelco.

9.8.1 PCB QC Check Solution (LCS Solution) - This solution is used for samples requiring PCB analysis also. The solution consists of Aroclor 1260 at a concentration of 10 ug/ml. The solution must be stored at 4□C (±2□C) in a Teflon sealed amber container for a maximum of 6 months. Source: Supelco.

10.0 CALIBRATION - N/A

11.0 QUALITY CONTROL

11.1 A minimum of one blank (consisting of a liter of reagent water spiked with surrogates and carried through the entire analytical procedure) will be extracted every day samples are processed. A maximum of 20 samples can be extracted under a method blank in a calendar day.

11.2 A matrix spike/matrix spike duplicate will be extracted at a frequency of 1 per case or 20 field samples in an SDG. However, if more than 7 days elapses and 20 samples have not been extracted, it will be necessary to extract an MS/MSD.

11.3 All logbooks and forms will be dated and filled out with black ink, and any errors must be crossed out with a single black line. The change must be signed and dated by the person making the change. In no case will the original entry be made unreadable. Any unused portion of the logbook page will be "Z"ed out. The logbook will be reviewed and approved periodically by the Group Leader or his/her designee.

11.4 Any solvents used must come from lots of solvent approved by the corporate QA Officer using the procedures established in the "Corporate SOP for Solvent Approval".

11.5 Any problems encountered in the course of analysis (i.e. extracts going dry, extract spillage, improper pH, etc.) shall be documented by noting in the case narrative and by filling out a Correction Action Report and/or NCM. The filled out CAR will be forwarded to the Group Leader or his/her designee who will give it to the QA Officer. The QA Officer will give the CAR to the Project Manager who will contact the client for instructions on how to proceed.

11.6 All glassware used in processing samples must be cleaned in accordance with the "SOP for Cleaning Glassware".

11.7 The extractions Batch Approval Sheets must be dated and filled out on a daily basis. They are located on the back of the extractions logbook page. There is also space provided for extraction information to be included in the case narrative. A copy of the sheet is included as Figure 3. The sheets will be reviewed and approved periodically by the Group Leader or his/her designee.

11.8 The Extractions Logbook must be completely filled out on a daily basis for every batch extracted or concentrated.

11.9 The lot numbers of reagents used in the extraction and concentration must be recorded in the reagents section of the Extractions Logbook Page (Figure 4).

11.11 The Extractions refrigerator and balance will be checked daily using the "SOP for

Temperature Monitoring of Ovens, Refrigerators and Freezers".

11.12 All analysts will perform an initial demonstration of extraction proficiency. The documentation will be maintained by the QA Officer.

11.13 A QC check sample (LCS) will be extracted at the same frequency as the method blank.

12.0 SAMPLE PREPARATION

12.1 Chain of Custody Procedures

12.1.1 All analysts are responsible for maintaining sample custody in accordance with EPA guidelines as detailed in "Documenting Sample Removal from the Laboratory".

12.1.2 All samples must be signed out on the chain of custody forms located in sample control.

12.2 Water Sample Extraction/Concentration Procedure - Separatory Funnel

12.2.1 When the sample is brought to the laboratory from sample control, the extraction logbook for the batch is started. Figure 4 is an example of an Extraction Logbook Page. Each section of the figure has a letter in it designating who has responsibility for completing the section. The person extracting is designated by an "E", and the person doing the concentration is designated by a "C". Enter the appropriate sample and solvent lot information on the logbook page.

12.2.2 The pH of the properly mixed sample is checked using pH paper. If the pH is outside the range of 5-9, it is noted in the comment section of the logbook and LIMS. The pH is adjusted to be in the range of 5-9 using sulfuric acid or 10N sodium hydroxide. A few drops of the sample should be dropped onto the pH paper, not dipped into the sample bottle.

12.2.3 The chlorine residual of the sample is checked using the chlorine test kit/tabs.

a. For routine samples, the chlorine residual is neutralized using a 10% sodium thiosulfate solution, or sodium thiosulfate pellets. This is noted in the comments section of the logbook and LIMS.

b. For projects where neutralization of the chlorine residual is not an option, a second 1 L aliquot will be extracted concurrently with the original sample.

12.2.4 If the sample is submitted in a 1 liter bottle, the level of the sample is marked on the side of the bottle with an indelible marker so the volume can be determined with a 1 liter

graduated cylinder. The sample is mixed to ensure homogeneity, and transferred to a 2-liter separatory funnel. If the sample is submitted in a larger volume bottle, a pre-cleaned graduated cylinder is used to measure 1 liter of well-mixed sample. The appropriate information is entered into the extraction logbook.

a) If an MS/MSD is to be run, measure out two extra 1-liter aliquots for that sample.

b) If the sample is for TCLP, 200 mLs of sample will be used for the extraction.

12.2.5 With every batch of samples processed, a blank must be extracted. The blank will consist of 1 liter of reagent water spiked with surrogates and carried through the entire analytical procedure.

a) Additionally, for each batch of TCLP samples leached, a TCLP blank will be provided to extractions. The TCLP blank and TCLP blank QC will be treated as a sample by extractions.

12.2.6 To ensure that every extraction is properly surrogated and/or spiked, the process must be witnessed by another analyst. The witnessing analyst will also initial the extractions logbook.

12.2.7 Add 1,000 uL of pesticide surrogate to every blank, sample and spike using a 1 mL syringe.

12.2.8 If an MS/MSD is to be run with a batch, add 1,000 uL of the appropriate spike solution to the MS and MSD using a 1 mL syringe. The frequency of MS/MSD analysis is specified in Section 10.2.

a) For TCLP samples with client specified QC, it is only necessary to do an MS using 1,000 uL of the TCLP pesticide MS solution. NYSDEC, however, also requires an MSD using the TCLP pesticide MS solution for TCLP samples.

b) For NYSDEC, any time an MS/MSD is extracted it is also necessary to extract an MSB (matrix spike blank). Add 1,000 uL of the appropriate spike solution to a liter of reagent water spiked with surrogates. Call it "LabID MSB" (e.g. 1234001MSB). For TCLP samples use 1,000 uL of the TCLP pesticide MS solution.

12.2.9 For the QC check sample (LCS), add 100 uL of the pesticide QC check solution to a liter of reagent water. Call it "BlankID QC" (e.g. 112299-B01QC). For TCLP samples, use 100uL of the TCLP pesticide QC solution.

a) For NYSDEC TCLP samples with client requested QC, the MSB is the LCS since the MSB contains the full target list. This will also be true for other projects that require a full compound matrix spike.

b) For PCB's QC check (LCS) add 1,000 uL of the PCB QC check solution to a liter of reagent water.

12.2.10 Fill in the appropriate surrogate and spike volume information in the extraction logbook. Initial the appropriate extraction and spiking blocks in the extractions logbook.

12.2.11 Add 60 mL of methylene chloride directly to the separatory funnel. Extract the sample by shaking the funnel for 2 minutes with periodic venting to release the excess pressure. Allow the organic layer to separate from the water phase. If an emulsion forms at the interface between the 2 layers, and it is greater than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to break the emulsion.

a) Acceptable mechanical techniques for breaking an emulsion include: stirring, filtering the emulsion through glass wool, or centrifugation.

b) If the emulsion cannot be broken, it will be necessary to use a continuous liquid-liquid extractor on the sample. Details on the procedure can be obtained in the liquid-liquid extraction procedure in Section 11.3.

12.2.12 The organic layer is then collected in a labeled amber jar. It is permissible to dry the extract by passing it through a funnel containing sodium sulfate.

a) The label should include the sample number and the parameter analyzed. Colored tape is used for the label.

12.2.13 The extraction is repeated twice more, using fresh 60 mL portions of methylene chloride. The extracts are combined in an amber jar.

12.2.14 Assemble a K-D concentrator by attaching a 10 mL concentrator tube to a 500 mL evaporative flask. Transfer the tape label to the flask.

12.2.15 If the extract was not dried at the time of extraction, prepare a drying column by placing a plug of glass wool in the bottom of a powder funnel and filling it with approximately 20 g of anhydrous granular sodium sulfate. Rinse the column with methylene chloride, and discard the rinse.

12.2.16 Pour the combined extract through the drying column into the K-D concentrator. Rinse the amber jar with 20-30 mLs of methylene chloride to quantitatively transfer the sample.

12.2.17 Add one or two clean boiling chips to the K-D and attach a 3-ball snyder column. Pre-wet the snyder column with about 1 mL methylene chloride. Place the K-D in a hot water bath (80-85□C) so that the concentrator tube is partially immersed in the hot water bath and the

entire lower rounded surface of the flask is bathed with hot vapor. The temperature should be such that the concentration is completed within 20-25 minutes. When the apparent volume reaches 3-4 mLs, remove the K-D and add 60 mLs of hexane to the K-D through the snyder column, and concentrate as above. When the apparent volume reaches 3-4 mLs, remove from the batch and allow the K-D to cool.

a) At the proper rate of distillation, the balls in the column will actively chatter but the chambers will not flood with condensed solvent.

b) If GPC cleanup is required, remove the snyder column, rinse the flask and its lower joint into the receiving vessel and adjust the volume to 10 mLs with methylene chloride. Proceed with the GPC cleanup according to the latest version of the SOP for "GPC Cleanup of Pesticide Extracts Method 3640". After GPC cleanup, concentrate as above and proceed with the solvent exchange to hexane.

12.2.18 Remove the snyder column, and rinse the flask and its lower joint into the concentrator tube with hexane. Bring the extract to a final volume of 10 mLs with hexane in a receiving vessel. It may be necessary to reduce the volume by placing the concentrator tube in the N-Evap and evaporating the solvent using a gentle stream of clean, dry nitrogen. During the concentration step, the solvent level must be kept below the water level of the batch. The extract must never be allowed to go dry.

a) If the extract has undergone GPC cleanup, the final volume will be 5 mLs.

12.2.19 Transfer the extract to a labeled Teflon lined screw cap vial using a pasteur pipet.

12.2.20 Fill in the appropriate concentration spaces in the extractions logbook.

12.2.21 The PCB/Pesticide extract is now ready for analysis. Place the extracts in the appropriate refrigerator maintained at 4□C in the GC lab. Fill out the extract chain of custody (Figure 1).

12.3 Sample Extraction - Continuous Liquid-Liquid Extraction/Concentration

12.3.1 When the sample is brought to the laboratory from sample control, the extraction logbook for the batch is started. Figure 4 is an example of an extraction logbook page. Each section of the figure has a letter in it designating who has responsibility for completing the section. The person extracting is designated by an "E", and the person doing the concentration is designated by a "C". Enter the appropriate sample and solvent lot information on the logbook page.

12.3.2 Set up the continuous extractor. Add a few boiling chips to the distilling flask. Add methylene chloride to the bottom of the extractor so that the crossover tube is half filled

with solvent, and charge the distilling flask with additional methylene chloride to bring to a total of ~450 mLs.

12.3.3 The pH of the properly mixed sample is checked using pH paper. If the pH is outside the range of 5-9, it is noted in the comment section of the logbook and LIMS. The pH is adjusted to be in the range of 5-9 using sulfuric acid or 10N sodium hydroxide. A few drops of the sample should be dropped onto the pH paper, not dipped into the sample bottle.

12.3.4 The chlorine residual of the sample is checked using the chlorine test kit/tabs.

a. For routine samples, the chlorine residual is neutralized using a 10% sodium thiosulfate solution, or sodium thiosulfate pellets. This is noted in the comments section of the logbook and LIMS.

b. For projects where neutralization of the chlorine residual is not an option, a second 1 L aliquot will be extracted concurrently with the original sample.

12.3.5 If the sample is submitted in a 1 liter bottle, the level of the sample is marked on the side of the bottle with an indelible marker so the volume can be determined with a 1 liter graduated cylinder. The sample is mixed to ensure homogeneity, and transferred to a 2-liter separatory funnel. If the sample is submitted in a larger volume bottle, a pre-cleaned graduated cylinder is used to measure 1 liter of well-mixed sample. The appropriate information is entered into the extraction logbook.

a) If an MS/MSD is to be extracted with a batch, two extra 1 liter aliquots are measured out.

b) If the sample is for TCLP, 200 mLs of samples will be used.

12.3.6 With every batch of samples processed, a blank must be extracted. The blank will consist of 1 liter of reagent water spiked with surrogates and carried through the entire analytical procedure.

a) Additionally, for each batch of TCLP samples leached, a TCLP blank will be provided to extractions. The TCLP blank will be treated as a sample by extractions.

12.3.7 To ensure that every extraction is properly surrogated and/or spiked, the process must be witnessed by another analyst. The witnessing analyst will also initial the extractions logbook.

12.3.8 Add 100 uL of pesticide surrogate to every blank, sample and spike using a 100uL syringe.

12.3.9 If an MS/MSD is to be run with a batch, add 1,000 uL of the appropriate matrix spike solution to the MS and MSD using a 1 mL syringe. The frequency of MS/MSD analysis is specified in Section 10.2.

a) For TCLP samples with client specified QC, it is only necessary to do an MS using 1,000 uL of the TCLP pesticide MS solution. NYSDEC SW846, however, also requires an MSD using the 1,000 uL of the TCLP pesticide MS solution for TCLP samples.

b) For NYSDEC, any time an MS/MSD is extracted it is also necessary to extract an MSB (matrix spike blank). Add 1,000 uL of the appropriate matrix spike to a liter of reagent water spiked with surrogates. Call it "LabID MSB" (e.g. 1234001MSB). For TCLP samples use 1,000 uL of the TCLP pesticide MS solution for the MSB.

12.3.10 For the Pesticide QC check sample (LCS), add 100 uL of the pesticide QC check solution to a liter of reagent water. Call it "BlankID QC" (e.g. 112299-B01QC). For TCLP samples, use 1 00 uL of the TCLP pesticide QC solution. The frequency of LCS analysis is stated in Section 10.15.

a) For NYSDEC SW846 TCLP samples with client requested QC, the MSB is the LCS since the MSB contains the full target list. This will also be true for other projects that require a full compound matrix spike.

b) For the PCB QC check sample (LCS) add 1000 uL of the PCB QC check solution to a liter of reagent water.

12.3.11 Fill in the appropriate surrogate and spike volume information in block 5 of the extraction logbook. Initial the appropriate extraction and spiking blocks in the extractions logbook.

12.3.12 Connect the distilling flask to the extractor body, and put the condenser on the top of the extractor body.

12.3.13 Turn on the chiller, and turn the heating mantles to approximately 3.5. Note the time boiling starts, and extract for 18 hours. Periodically check the extractor to ensure that the methylene chloride is cycling over.

12.3.14 At the end of the 18 hour extraction period, turn off the heating mantles and allow to cool. Turn the chiller off and transfer the methylene chloride remaining in the bottom of the extractor body to the distilling flask.

12.3.15 Assemble a K-D concentrator by attaching a 10 mL concentrator tube to a 500 mL evaporative flask. Transfer the tape label to the flask.

12.3.16 Prepare a drying funnel by placing a plug of glass wool or filter paper in the bottom of the funnel and filling it approximately halfway with anhydrous granular sodium sulfate. Rinse the funnel with methylene chloride.

12.3.17 Pour the extract through the drying funnel into the K-D concentrator. Rinse the distilling flask with 20-30 mLs of methylene chloride to quantitatively transfer the sample.

12.3.18 Add one or two clean boiling chips to the K-D and attach a 3-ball snyder column. Pre-wet the snyder column with about 1 mL methylene chloride. Place the K-D in a hot water bath (80-85□C) so that the concentrator tube is partially immersed in the hot water bath and the entire lower rounded surface of the flask is bathed with hot vapor. The temperature should be such that the concentration is completed within 20-25 minutes. When the apparent volume reaches 3-4 mLs, remove the K-D and add 60 mLs of hexane to the K-D through the snyder column, and concentrate as above. When the apparent volume reaches 3-4 mLs, remove from the batch and allow the K-D to cool.

a) At the proper rate of distillation, the balls in the column will actively chatter but the chambers will not flood with condensed solvent.

b) If GPC cleanup is required, remove the snyder column, rinse the flask and its lower joint into the receiving vessel and adjust the volume to 10 mLs with methylene chloride. Proceed with the GPC cleanup according to the latest version of the SOP for "GPC Cleanup of Pesticide Extracts Method 3640". After GPC cleanup, concentrate as above and proceed with the solvent exchange to hexane.

12.3.19 Remove the snyder column, and rinse the flask and its lower joint into the concentrator tube with hexane. Bring the extract to a final volume of 10 mLs with hexane in a receiving vessel. It may be necessary to reduce the volume by placing the concentrator tube in the N-Evap and evaporating the solvent using a gentle stream of clean, dry nitrogen. During the concentration step, the solvent level must be kept below the water level of the batch. The extract must never be allowed to go dry.

a) If the extract has undergone GPC cleanup, the final volume will be 5 mLs.

12.3.20 Transfer the extract to a labeled Teflon lined screw cap vial using a pasteur pipet.

12.3.21 Fill in the appropriate concentration spaces in the extractions logbook.

12.3.22 The PCB/Pesticide extract is now ready for analysis. Place the extracts in the appropriate refrigerator maintained at 4□C in the GC lab. Fill out the extract chain of custody and note the location in the extraction logbook.

13.0 CALCULATIONS - N/A

14.0 ACCEPTANCE OF DATA - N/A

15.0 REPORTING OF RESULTS

15.1 Copies of logbook pages, any CAR's and GPC chromatograms will be put in the client job folder.

16.0 SUPPLEMENTAL DOCUMENTS

16.1 The analyst is referred to other extractions SOPs as well as other departments SOPs for additional information concerning sample storage, sample removal, sample tracking and other information not included in this SOP.

17.0 REFERENCES

17.1 USEPA SW846 - Test Methods for the Evaluation of Solid Waste, 3rd Ed.

18.0 POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

18.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

18.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

18.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

18.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

18.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

18.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions

19.0 WASTE MANAGEMENT

The following waste streams are produced when this method is carried out.

- Extracted aqueous samples contaminated with methylene chloride. This material must be neutralized before it is discharged to a POTW. The aqueous waste is poured through a funnel containing sodium bicarbonate. This waste is then neutralized with Sulfuric acid before being discharged.
- Used sodium sulfate and glass wool or filter paper contaminated with methylene chloride from the extract drying step. This waste is collected in day containers labeled for non-regulated solid waste. This waste is then transferred daily to the large waste drum labeled for non-regulated solid waste in the Waste Storage room .
- Assorted flammable solvent waste from various rinses. This waste is collected in a 2 liter beaker labeled for mixed waste. When this is full it is poured in a day container labeled for mixed waste. This is transferred daily to a large waste drum labeled for mixed waste in the Waste Storage room.
- Methylene chloride waste from various rinses. Methylene chloride waste is collected in a labeled 2 liter beaker then disposed of in the waste drum labeled for methylene chloride waste in the in the Waste Storage room.

Miscellaneous disposable glassware contaminated with acids, caustics, solvents and sample residue.

All waste shall be managed in accordance with all state and federal requirements. See the TestAmerica-CT RCRA Contingency Plan.

19.2 All personnel who handle or generate waste must be trained within six months of employment in proper waste handling and requirements.

20.0 SUBSTANTIVE REVISIONS

20.1 Original issue - 09/15/93.

20.2 Changed name from IEA to AEN, removed SOP reference codes - 10/15/97.

20.3 Changed name from AEN to STL, Update method references- 2/02/99.

20.4 Revised section 7.3, receiver calibration, added sections 8.7.1 and 8.8.1 - PCB solutions, revised section 22.2 and 11.3 to include separate PCB spiking solutions; revised section 8.1 to change the method blank criteria; 03/05/99.

20.5 Added sections on terms & definitions, pollution prevention and waste management, renumbered sections affected by the additions, along with minor revisions to reflect current practices and added statement regarding water temperature to the receiving vessel calibration requirements - 4/3/02 (HR).

20.6 Revised Environmantal Health and Safety sections to reflect current practice. 5/21/04 WDG.

20.7 Minor revisions to reflect current practices. Updated pesticide QC amount to reflect what is currently being used.06/04/07 SBW

20.8 Added source of surrogates and spikes. Added new TestAmerica SOP header and control number, updated waste disposal; 10/26/07 sbw

TABLE 1.0

PESTICIDE QC CHECK (LCS) AND FULL MATRIX SPIKE, UG/ML

Analyte	Stock	Standard for Extractions
Aldrin	1.0	0.20
Dieldrin	1.0	0.20
p,p'DDT	5.0	1.0
p,p'DDE	1.0	0.2
p,p'DDD	5.0	1.0
Endosulfan I	1.0	0.20
Endosulfan II	5.0	1.0
Endosulfan Sulfate	5.0	1.0
Endrin	5.0	1.0
Endrin Aldehyde	5.0	1.0
Heptachlor	1.0	0.20
Heptachlor Epoxide	1.0	0.20
alpha-BHC	1.0	0.20
beta-BHC	1.0	0.20
gamma-BHC	1.0	0.20
delta-BHC	1.0	0.20
Methoxychlor	10.0	2.0
Endrin Ketone	5.0	1.0
4,4'DDT	1.0	0.20

Title: SOP for Pesticides
[Method 8081A]

Approvals (Signature/Date):

Technical Manager	Date	Health & Safety Manager / Coordinator	Date
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. GCS02208.CT.

Any reference within this document to Severn Trent Laboratories, Inc. or STL, should be understood to refer to TestAmerica Laboratories, Inc. (formerly known as Severn Trent Laboratories, Inc.)

Facility Distribution No. _____ Distributed To:_____

1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 This method defines the specific steps for analyzing and determining the concentration of various organochlorine pesticides in multimedia, multi-concentration samples.

2.2 It is the policy of TestAmerica and of the chromatography group to ensure that we administer contracts and orders for goods and services in a manner that is fully compliant with governmental laws and regulations, as well as the TestAmerica Policy Statement on Business, Ethics and Conductivity.

2.3 Refer to Table 6 of this SOP for the list of parameters.

2.4 The document control number for this SOP is GCS02208.CT.

3.0 TERMS AND DEFINITIONS

3.1 There are many definitions used with in the laboratory, which may be generic to all laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used with in the laboratory, reference the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 This method outlines the gas chromatographic procedure for the detection of organochlorine pesticides. Samples are extracted using the proper extraction technique. The extracts are analyzed by gas chromatography with an electron capture detector.

4.2 This SOP is based on the following methods:

- EPA Method 8081A (Organochlorine Pesticides by Gas Chromatography)
- EPA Method 8000B (Gas Chromatography)
- EPA Method 8000C (Gas Chromatography)

4.3 Deviations to Method

4.3.1 A modification is that our QC check solution does not routinely contain multi-component pesticides due to peak interferences between the multi-component chromatograms and single-component peaks.

4.3.2 Toxaphene or Technical chlordane QC checks are prepared if required by for a specific project.

5.0 INTERFERENCES

5.1 Phthalate esters can interfere with pesticide determination; avoid any contact with plastics to best minimize this problem.

5.2 Sulfur is also an interference; this can be removed by performing sulfur clean-up on the extract. When using mercury, refer to the Pesticide Extract Sulfur Removal SOP.

6.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

6.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

6.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

7.1 Sample Containers

- Water samples are collected in 2x1 liter amber glass containers with Teflon-coated liner and Telfon coated caps.
- Soil samples are collected in 250 or 500 mL glass containers with Teflon coated caps.
- Sample bottles are never reused.

7.2 Sample Collection

- Samples are secured against breakage in the shipping containers and kept at 4°C for transport to the laboratory. Samples should arrive at the laboratory the next day following collection.

7.3 Sample Preservation

- Samples are preserved by cooling to 4°C.

7.4 Holding Times

- Water samples must be extracted within seven days from collection.
- Soil samples must be extracted within 14 days from collection.
- Aqueous samples requiring NYSDEC ASP must be extracted within five days from VTSR.
- Soil samples requiring NYSDEC ASP should be extracted within 14days from collection.
- All extracts must be analyzed within 40 days from date of extraction (40 days from collection for NYSDEC ASP).

8.0 APPARATUS AND MATERIALS

8.1 Sample extracts are analyzed on a gas chromatograph (GC) equipped with an electron-capture detector (ECD), autosampler, data collection system and all other required accessories. The following columns are acceptable for analysis:

Reference Sect 12.1 for Instrument specifications

- Restek Rtx-CLPesticides 30 meter
  0.53mm ID 0.42um film thickness
  or equivalent
- Restek Rtx-CLPesticidesII 30meter
  0.53mm ID 0.42um film thickness
  or equivalent
- Various sizes of syringes, volumetric pipets, volumetric flasks, pipet bulbs
- 0.8 ml and 1.8 ml autosampler vials and caps.

- Borosilicate glass transfer pipets/transfer bulbs.
- Safety glasses, non-powdered polyvinyl gloves, fume hood.
- Properly cooled refrigerators each for sample and standard storage.
- Standard and Instrument Maintenance logbooks.
- Sample Injection logbooks.

9.0 REAGENTS AND STANDARD PREPARATION

9.1 **Solvents**: Hexane, Acetone, Toluene and Isooctane (2,2,4-trimethylpentane) should be pesticide grade or equivalent.

9.2 Commercially prepared stock standards can be used if they are certified and pretested by the manufacturer.

9.3 All stock and working standards are stored in amber screw top bottles at 4°C and replaced after 1 year or earlier if routine QC tests indicate a problem.

9.4 Calibration Stock Standard

9.4.1 Individual Stock Mix A and B

Calibration Stock Standard AB is prepared by diluting the standard mix, purchased from a commercial vendor, into isooctane. See Table 1.0 for individual compounds and concentrations. See Table 2.0-2.2 for Preparation of Mixes. When there is insufficient resolution between compounds, Mix A and B may be prepared separately to create two sets of standards of non-coeluting compounds to be run separately.

9.5 **Single-component Calibration Standards** at a minimum of 5 concentration levels are prepared through dilution of the calibration stock standard, with a lower 6th level (designated as level 0.5) used as needed for lower reporting limits. One concentration level should be near but above the method detection limit. The remaining concentrations should correspond to the linear range of the instrument. See Tables 1.0 for final component concentrations.

9.5.1 For Army Corp. Projects the lowest calibration level should be near but above the MDL and never less than three times the MDL.

9.6 **Pesticide Multi-component Standards** - A single midpoint calibration standard is prepared for Toxaphene and Technical Chlordane as required. Each of these standards are diluted from individual stocks. Each is run after a single component curve. For Multi-component Stock and Standard concentrations see Table 1.0. For Multi-componenet standard preparations see Table 2.3-2.4.

9.6.1 For Army Corp. Projects: If Toxaphene or Technical Chlordane is detected in a sample; the sample will be reanalyzed against a calibration curve of at least 3 levels.

9.7 **Surrogate Standard** is prepared to monitor the performance of the extraction and analytical system. Samples, standards and blanks are spiked with pesticide surrogates. Two surrogates, 2,4,5,6-tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB), are spiked with the proper amount at a level of 0.2 ug/L for waters and soils are spiked at a level of 6.7 ug/Kg. (See Table 3.0).

9.8 **Instrument Breakdown Standard** is prepared as a solution of p,p'DDT and Endrin at a concentration near the midlevel concentration of the calibration standards. (See Table 5.0).

10.0 CALIBRATION

10.1 RT Windows are established by making three injections of the mid concentration standard throughout the course of a 72-hour period and calculating 3 x ± the standard deviation. Retention time windows shall be calculated for each compound on each GC column whenever a new column is installed.

10.1.1 RT windows are established as described above, however, the laboratory has established a minimum RT window of 0.03minutes.

10.2 Calibration Standards

Initial calibration standards are analyzed by injecting 1 ul of each of the levels of the single component calibration standards Mix A & Mix B, and one level of all other multi-response pesticides/PCB's

10.2.1 The initial calibration criteria is 20% RSD for calibration factors form the intial calibration standards for each target analyts. If the %RSD cannot be met using average calibration factor, an alternate curve type can be used. A Linear regression fit requires a minimum of 5 points and a Quadratic fit requires a minimum of 6 points. The criteria is 0.990 or higher for the coefficient. In determining the best fit of a curve, using either linear regression or Quadratic, the analyst shall choose the best fit possible based on both the coefficient and the slope of the line (b). The coefficient should be greater than 0.990 and as close to 1 as possible. The slope of the line (b) should be as low a value as possible. The analyst may

choose between linear regression, a weighted linear regression or a Quadratic fit. The line may be forced through zero, if the (b) value by other means would result in possible negative results.

10.3 The working calibration range of this method is defined by the initial calibration curve. All extracts with target compounds exceeding the curve should be diluted to within the upper half of the calibration range.

10.3.1 The daily calibration verification is acceptable if all of the following are true.

10.3.1.1 The breakdown of Endrin or p'p'DDT is <= 15% based on the presence of Endrin Aldehyde and Endrin Ketone for Endrin, p,p'DDD and p,p'DDE for p,p'DDT. If the breakdown does exceed 15% then corrective action must be taken prior to continuing the calibration verification.

$$\% \text{ breakdown for p'p'DDT} = \frac{\text{DDD peak area + DDE peak area}}{\text{Total DDT peak area (DDT+DDD+DDE)}} \times 100$$

% breakdown for Endrin =

$$\frac{\text{Endrin Aldehyde peak area + Endrin Ketone peak area}}{\text{Total Endrin peak area (Endrin+Endrin Aldehyde+Endrin Ketone)}} \times 100$$

10.3.1.2 The calibration verification standard must have all compounds (+/-) 15% difference of their expected value. Because of the low concentration of pesticide standards injected on a GC/EC, column adsorption may be a problem when the GC has not been operated for a day. Therefore, the GC column should be primed by injecting a pesticide standard mixture approximately 20 times more concentrated than the midlevel standard.

If the calibration verification standard fails, then it can be rerun. A new calibration curve will be run, if warranted, due to repeated failure. When the calibration verification standard fails, all samples that were injected after the last standard that met the QC criteria must be evaluated to prevent any mis-quants and possible false negatives. Depending on the compound failures the extracts may need reinjection. More frequent analysis of standards will minimize the number of reruns for QC failures.

If the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e. >15%, and the analyte was not detected in the specific sample analyzed during the sequence, then the sample extracts do not need to be reanalyzed. If the compound was detected in the sample and the sample bracket is 10 or less, the chromatography is reviewed by the department supervisor for the possibility of sample matrix. No further action may be taken at this point.

10.3.1.3 An instrument blank standard is run after the calibration verifications standards and prior to any samples to ensure the instrument and its autosampler are clean.

10.3.2 All initial calibrations must be verified with a standard obtained from a second source manufacturer or lot. The same manufacturer may be used, if the lot can be demonstrated from the manufacturer as prepared independently from other lots. Traceability shall be to a national standard when commercially available. The % difference of this standard should pass within +/- 25% from the initial calibration curve. If the second source standard fails to meet criteria, check the preparation of both the initial curve and the second source standard for errors. It may be necessary to re-prepare a solution to check.

10.4 When analyzing samples, any extract that contains a target compound that exceeds the high level calibration must be diluted to within the calibration range. Multi-component targets must also be diluted so that the largest peak in the multi-component does not exceed the response of the largest peak in the high-level single-component calibration standard.

10.5 Calibration Verification - (Each 12 hour shift)

10.5.1 A single mid-level standard (standard mix or multi-compound), is analyzed at a minimum of every 20 samples and at the end of the analysis sequence. If the samples are suspected to cause breakdown and column degradation the calibration check will be analyzed every 10 samples. The calibration factor for each compound to be quantitated, must not exceed a 15 percent difference when compared to the average calibration factor from the calibration curve. When this criteria is exceeded, inspect the GC system to determine the cause and perform whatever maintenance is required before recalibrating and proceeding with sample analysis. All samples that were injected prior to the standard exceeding the criteria must be reinjected if the initial analysis indicated the presence of the specific target analytes that exceeded the criteria.

10.5.2 For Army Corp. Projects a mid-level calibration verification must be analyzed every 10 samples and at the end of the analysis sequence.

10.6 Calculations for water samples:

10.6.1 The calculation for quantitation of single-component target analytes is as follows:

Single Component Calculation

$$ug/L = \frac{\text{Area of sample peak x Final Volume of extract(uL) x Dilution Factor}}{\text{Avg Calibration Factor of standard x Sample Volume extracted (mLs) x ul inj.}}$$

$$CF = \frac{\text{Peak Area of the Standard}}{}$$

Mass Injected (ng)

Water sample by Linear regression:

b + (Area) m1 = on column amount (ng)

(cn column amount-ng) (final volume [uL])(dilution factor) = ug/L
(sample volume [mLs])(ul inj.)

Water sample by Quadratic:

b + m1(Area) + (m2)*(Area)^2 = on column amount (ng)

(cn column amount-ng) (final volume [uL])(dilution factor) = ug/L
(sample volume [mLs])(ul inj.)

10.6.2 An average calibration factor is calculated for each compound and surrogate from the initial calibration. For multi-component analytes a calibraton factor is calulated for the areas of each peak (using 3 -5). The final concentration is then calculated using the average of the final result for each of the 3 - 5 peaks.

The peaks chosen for quantitation should be major peaks representative of the multicomponent standard. The areas of each peak should be evaluated in relation to each other and any disproportionate peaks should not be used.

Multicomponent Calculation

ug/L = (X) Final Volume of extract (uL) x Dilution Factor
(Y) Sample Volume extracted (mLs) x ul inj.

X = The area of one of the 3-5 peaks from sample.
Y = The average CF for the corresponding peak from standard.

A Cf must be calculated for 3-5 peaks. The final concentration reported is calculated using the average of the final result for each of the 3-5 peaks.

10.7 Calculations for soil and oil samples:

10.7.1 The calculation for quantitation of single-component target analytes is as follows:

Single Component Calculation

ug/Kg = Area of sample peak x Final Volume of Extract (uL) x Dilution Factor
Avg. Calibration Factor of Std. x Sample Volume Extracted(g) x Decimal % Solids x ul inj.

$$CF = \frac{\text{Peak Area of the Standard}}{\text{Mass Injected (ng)}}$$

10.7.2 An average calibration factor is calculated for each compound and surrogate from the initial calibration. For multi-component analytes a calibraton factor is calulated for the areas of each peak (using 3 -5). The final concentration is then calculated using the average of the final result for each of the 3 - 5 peaks.

The peaks chosen for quantitation should be major peaks representative of the multicomponent standard. The areas of each peak should be evaluated in relation to each other and any disproportionate peaks should not be used.

Multicomponent Calculation

$$ug/Kg = \frac{(X) \text{ Final Volume of Extract (uL)} \times \text{Dilution Factor}}{(Y) \text{ Sample Volume Extracted(g)} \times \text{Decimal \% Solids} \times \text{ul inj.}}$$

X = The area of one of the 3-5 peaks from sample.
Y = The average CF for the corresponding peak from standard.

A Cf must be calculated for 3-5 peaks. The final concentration reported is calculated using the average of the final result for each of the 3-5 peaks.

Note: Oils will not have decimal percent solids.

10.7.3 When more than one multicomponent is detected in a sample, non-overlapping peaks are chosen for quantitation. If it is not possible to choose non-overlapping peaks, peaks with the least amount of overlap are chosen.

10.8 Corrective Action for Initial Calibration

10.8.1 If the technical acceptance criteria for the initial calibration are not met, inspect the system for problems. It may be necessary to change the column, bake out the detector, clean the injection port, or take other corrective actions to achieve the acceptance criteria.

10.8.2 Corrective Action for Calibration Verification

10.8.3 If the technical acceptance criteria for the calibration verification are not met, inspect the system for problems and take corrective actions to achieve the criteria.

10.8.3 Major corrective actions such as replacing the column or detector will required a new initial calibration.

10.8.4 Minor corrective actions may not require a new initial calibration provided a calibration check meets all acceptance criteria.

11.0 QUALITY CONTROL

11.1 Demonstration of Analyst Capability-Spike four sets of reagent using the Pesticide QC solution. Process the samples through the whole analytical procedure.

11.1.1 Calculate the average recovery (x) and the standard deviation (s) for each analyte from the four results. Compare the s and x with the criteria generated from the laboratory control charts for each compound for each matrix. The limits are derived from laboratory-generated data, and are updated as needed. If all analytes meet the acceptance criteria, analysis of samples can begin. If any analyte fails, the cause for the failure must be determined and the test must be repeated for that analyte.

11.1.2 The demonstration of analyst capability will be verified on an annual basis.

11.2 Method detection limits (MDL's) for this method will be verified on an annual basis as detailed in the latest version of the corporate SOP on MDL's. The MDL check sample is an extracted sample containing each target analyte at a concentration close to the MDL. Each analyte must be detected in order for the instrument to be considered capable of reporting estimated result to the calculated MDL. If an analyte cannot be detected at the given concentration, instrument maintenance should be performed and the MDL check sample re-analyzed. If the compound is still not detected, a new MDL should be prepared and analyzed at a higher concentration. The new MDL should be used in reporting results.

11.3 External PT samples are randomly submitted by the QC officer and are processed as any other client's samples would be.

11.4 Refer to Table 6.0 for Practical Quantitation Limits (PQL's) for all compounds.

11.5 Matrix spike (MS), matrix spike duplicates (MSDs) and matrix spike blanks (MSB's), if applicable, are analyzed within every set of 20 samples or less. Recoveries must be within the laboratory generated control limits. If these criteria are not met, but the blank spike data meet all of the recovery criteria, then the MS/MSD are documented as having matrix interferences. If the blank spike fails, check for instrument and/or column related problems and reanalyze the spikes. If the problem is corrected, the samples are reanalyzed.

11.6 A QC check sample is performed at a frequency of one per 20 samples extracted of similar matrix. See Table 7.0 for spike compounds. Control charts are used to establish laboratory generated control limits.

11.7 Blanks

11.7.1 Method Blank

Method blanks are spiked with surrogates, extracted, and analyzed following the same procedure that is used with the associated samples. A water method blank is one liter of reagent water and a soil method blank is 30 grams of sodium sulfate for sonication and 15 grams of sodium sulfate for automated soxtherm extraction.

Frequency - method blanks must be analyzed with each case, 20 samples of a similar matrix, or whenever samples are extracted by the same procedure, whichever is more frequent.

Acceptance criteria - method blanks must contain less than half the PQL for all the target compounds listed in Table 1.0 & 1.1.

All samples associated with an unacceptable method blank must be reextracted and reanalyzed.

11.8 Surrogates

11.8.1 The surrogates TCX and DCB are added to each standard, sample, blank and QC prior to extraction.

11.8.2 The QC limits for surrogate recovery are listed in Table 3.1 and pertain to all samples, blanks and spikes.

11.9 Analytical QC Samples

Daily Calibration Check Sample	One per 12 hour shift minimum
Breakdown Standard	One per 12 hour shift minimum
Instrument Blank	One per 12 hour shift minimum

*It may be necessary to analyze a solvent blank after high concentration samples.

Preparation QC Samples

MS/MSD	One pair every 20 samples minimum
QC Check	Every extraction batch
Surrogates	Every sample and standard
Method Blank	One per batch

11.10 Analytical Documentation Procedures

11.10.1 Instrument Batches

An instrument batch is created for each analytical sequence to organize all the associated data. Batch designations are of the format:

XXnnn

where: XX = instrument identifier
nn = number of batch

(i.e. C5001)

Instrument batches are number sequentially so a unique batch identifier identifies each analytical sequence. The batch consists of a file folder with all the associated QC information for the analytical sequence. The raw data is then scanned for all initial and continuing calibrations.

11.10.2 Data Archiving

All data files are archived on a daily basis using a 12.0gb data storage cartridge. The associated method files are also archived daily to provide an accurate historical record. Care shall be exercised when purging data off the hard drives to ensure that all data being purged has been archived.

11.10.3 Instrument Run Logs

It is STL's policy that all measurement data be recorded in logbooks or on preprinted log sheets in permanent ink. Run logs are created from the Target data system by generating a file which contains a sequential list of all files analyzed. The record shall reflect the measurement performed and all appropriate details for conclusions related to the measurement. The record shall be signed and dated by the individual performing the measurement on the day the measurement is performed. Corrections shall be made by drawing a single line through the error, and initialing and dating the correction. A secondary authorization of the logbook is required and shall be performed by the department's manager or designee.

Each instrument has its own set of bound run logs (see Figure 1.0) which are sequentially numbered and paginated. Run logs are filed in the laboratory once they have been filled, for future reference. Each analytical sequence shall be started on a new page of the log and continued on the next page, if necessary. The header information designating the standard codes used shall be completed for each sequence. All standards used are recorded in this field for future traceability. The data file, sample number, dilution factor, analyst's signature, and date are recorded.

11.10.4 Corrective Action Reports

A corrective action report (CAR) is initiated when a problem is encountered during analysis, data reduction or deliverables preparation, data validation, or when any deviations from this SOP occur. The CAR is initiated by the analyst or dept. manager first identifying the problem through the NCM module in the LIMS system. It is then electronically forwarded to all appropriate departments, QA officer, and Lab Manager. Reference SOP for correction action reports.

11.10.5 Chain of Custody Record

When samples are removed from storage for preparation or analysis they must be signed out utilizing the chain of custody record (COC). The samples shall then be signed back in on the COC upon their return to storage or designated "used" if the sample volume is consumed during the preparation or analysis.

11.10.6 Sample Tracking Record

Notification of sample arrival is done by the Sample Control department by issuing a preliminary notification sheet. Samples are tracked for extraction and analysis by using the laboratory's LIMS system.

11.11 Quality Control Check Points

11.11.1 Analysis quality control approval report

Specific quality control checkpoints have been established for the analysis of sample extracts. The specific check points in the analysis logbook, are initialed and dated by the analyst to ensure the consistency and accuracy of the data produced.

11.11.2 Specific quality control checkpoints have been established for the preparation of data deliverables, which are monitored through a Lims Organics Data Review Checklist – Doc # QAF04300.CT. The specific check points must be reviewed by the analyst 1st level reviewing and the secondary reviewer 2nd level revieweing the data to ensure the consistency and accuracy of the data produced. Refer to Figure 3.0 for the document and specific control points covered.

12.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

12.1 HP6890 GC's with dual micro ECD's and CTC (Leap Technologies) Autosampler is currently being used to run Pesticides/PCBs under this SOP. Instruments, columns and

conditions following are guidance:

a) Column type: 30 meter Rtx-CLPesticides (See Section 7.1)
Column flow: ~ 5 mL/min Hydrogen or Helium
Detector Make-up Flow: 60° mL/min N_2
Injector Temperature: 225 C
Detector Temperature: 330°C
Temperature Programming:
Initial Temperature: 110°C Hold 1.0 min
Initial Ramp: 20°/min to 245, 0min Hold
Second Ramp: 6/min to 310, no hold

b) Column type: 30meter Rtx-CLPesticides II (See Section 7.1)
Oven and temperatures same as above

Instrument and column conditions may have equivalent programming as long as all method QC requirements are met.

12.2 **Aqueous Sample Preparation for Pesticides:**

12.2.1 Brief Summary:

A 1 liter sample aliquot is spiked with the surrogate and extracted with methylene chloride at a pH between 5 and 9. Using either the separatory funnel extraction method or the continuous liquid-liquid extraction method is acceptable. The methylene chloride extract is dried and solvent exchanged to hexane and adjusted to a final volume of 10 mL. The sample extraction must be completed within 7 days of sample collection. Samples requiring NYSDEC ASP must be completed within seven days of VTSR.

A 1 uL aliquot of the sample extract is injected into the gas chromatograph (GC). The GC separates the compounds. Compound identification is performed by the comparison of GC retention times to those of known analytical standards. Quantitative analysis is performed by the comparison of compound peak height or peak areas to those of an analytical standard at a known concentration.

12.2.2 For TCLP samples, 800 mLs of reagent water is added to 200 mLs of the TCLP leached sample and the procedure would follow Section 11.2.1. See Table 9 for TCLP PQL's and target compounds.

12.2.3 Sample Extraction

Refer to STL Standard Operating Procedure for Method 3510C for the water extraction procedure.

12.3 Soil Sample Preparation for Pesticides

12.3.1 Brief Summary

A 30 g sample aliquot is spiked with surrogate and extracted with a 1:1 mixture of hexane/ acetone using the sonication extraction method. The hexane /acetone extract is dried and concentrated to a final volume of 10 mL. The sample extraction must be completed within 14 days of sample collection. A 1uL aliquot of the sample extract is injected into the gas chromatograph (GC). The GC separates the compounds. Compound identification is performed by the comparison of GC retention times to those of known analytical standards. Quantitative analysis is performed by the comparison of compound peak height or peak areas to those of an analytical standard at a known concentration.

12.3.2 A 15 g sample aliquot is spiked with surrogate and extracted with a 1:1 mixture of hexane/ acetone using the soxtherm apparatus. The hexane /acetone extract is dried and concentrated to a final volume of 5 mL. The sample extraction must be completed within 14 days of sample collection. A 1uL aliquot of the sample extract is injected into the gas chromatograph (GC). The GC separates the compounds. Compound identification is performed by the comparison of GC retention times to those of known analytical standards. Quantitative analysis is performed by the comparison of compound peak height or peak areas to those of an analytical standard at a known concentration.

12.3.3 Sample Extraction

Refer to the STL Standard Operating Procedure for method 3550B and 3541 soil extraction procedures.

12.4 Oil Sample Preparation for Pesticides

12.4.1 Brief Summary

A 1 gm sample aliquot is spiked with surrogate and brought to a final volume of 10 mLs in hexane or other appropriate solvent.

12.4.2 If the sample is a TCLP oil, a 1 mL sample aliquot is spiked with surrogate and brought to a final volume of 10 mLs in hexane or other appropriate solvent.

12.5 Sample Analysis Procedures

12.5.1 Sample Extract Analysis

Sample extracts are removed from storage in the G.C. instrument room and are signed out

on the extract chain of custody form. All sample extracts are signed back in after they are returned to storage.

Make sure all instrumental operating conditions are correctly set.

In a vial with a 200 ul insert, load a 200 ul aliquot of sample extract. A 1.0 ul injection of the sample extract onto the GC column is made with an autosampler and then the GC temperature program sequence is started.

This method is intended to achieve the quantitation limits whenever possible. If sample chromatograms have interfering peaks, high baseline, or off-scale peaks, then those samples must be reanalyzed following dilution, cleanup, or reextraction. No limit is placed on the number of reextractions of samples that may be required because of contaminated method blanks.
The sample must be analyzed at the most concentrated level that is consistent with achieving satisfactory chromatography (defined in Section 12.7).

No target analyte concentrations may exceed the upper limit of the initial calibration.

12.6 Qualitative Analysis

12.6.1 Target Compounds

The identification of single component pesticides is based primarily on retention time data. The RT of a peak can be verified only from an on-scale chromatogram.

If a compound falls within the R.T. windows of the compound in the calibration curve, and is greater than the MDL, that sample would require confirmation by analysis on a second column.

12.6.2 If the same compound falls within the R.T. window of the compound on the column used for confirmation, the compound is determined to be present and therefore reported as a target compound. This second column should be of a dissimilar phase from the first column, see section 8.1.

12.7 Quantitative Analysis

12.7.1 Target Compounds

Target compounds are quantitated by the external standard technique using peak area and the calibration factor determined during the initial calibration sequence.

A second column confirmation analysis is performed on all samples for analytes with concentrations greater than the MDL.

When compound concentrations are below the PQL, but the compound meets identification criteria, report the concentration with a "J" qualifier.

When a compound exceeds the linear working range of the initial calibration, the sample must be diluted to bring the analyte concentration within the calibration range.

The following are guidelines on performing dilutions and exceptions to this requirement:

- If the response is still above the high calibration point after the dilution of 1:100,000, the laboratory shall contact the client.

- Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range.

12.7.2 The lab routinely reports the higher concentration of the results between the two columns used for analysis. If there is a greater than 40% difference between the two columns, the lower result is reported.

12.8 Instrument Maintenance

12.8.1 Preventative Maintenance

All instrumentation is covered by a service contract with an external instrumentation service vendor, or by STL personnel trained in preventative maintenance. All instrument preventative maintenance is performed according the manufacturers recommended procedures, by trained personnel. All preventative maintenance shall be thoroughly documented in the maintenance log (see Figure 4.0), as to a description of the maintenance performed, the date performed, and the personnel performing the maintenance.

12.8.2 Corrective Maintenance Determinants and Procedures

Corrective maintenance is deemed necessary when the analytical system, after reanalysis, cannot meet calibration, resolution, chromatography, breakdown, or other protocol specific QC criteria. Corrective maintenance may include, but is not limited to, decontamination of the system, injection port cleaning, column cutting or replacement, syringe cleaning or replacement, or detector baking out or replacement. All corrective maintenance is performed according the manufacturers recommended procedures, by trained personnel. All corrective maintenance shall be thoroughly documented in the maintenance log, as to a description of the maintenance performed, the date performed, and the personnel performing the maintenance.

12.8.3 Maintenance Authorization

The department's manager, or designee authorizes all preventative and corrective maintenance. When maintenance is deemed necessary, a service call is placed for all equipment covered under a service contract, by the department's manager, or designee.

12.9 Data System

12.9.1 Data Acquisition and System Operation

Data is acquired from sample analyses using Chemstation software. Analytical batches are set up with all the associated sample ID, dilution, and data file information. Raw data files are manually copied to the Target data system for integration and quantitation. Turbochrom has instrument control.

12.9.2 Instrument Errors

System errors are logged to the system console at time of occurrence. The system manager shall be responsible for checking and providing corrective actions for all system errors.

12.9.3 Manual Integration Editing Flags

Manual integrations shall be performed when the automated integration does not yield correctly integrated baselines. Manual integrations are flagged by the data system with the "M" qualifier beside the concentration of any manually integrated compound. The analyst name is printed on the quantitation report. If a name does not appear on the report, the analyst must manually date and sign the page.

13.0 CALCULATIONS

See Sections 10.5.1, 10.5.2, 9.6.1 and 10.6.2 of this document.

13.1 Calculation of Calibration Factor

$$CF = \frac{\text{Peak Area of the Standard}}{\text{Mass Injected (ng)}}$$

13.1.1 Calculation of Percent Difference

The following formula is used to calculate % difference in the calculated versus expected values of standards.

$$\% \text{ Difference} = \frac{\text{Calculated conc. - expected conc.}}{\text{Expected conc.}} \times 100$$

13.1.2 Calculation of Surrogate, Spike and QC Check Recoveries

The following calculation is used for spiked sample recoveries.

$$\% \text{ Recoveries} = \frac{\text{Amount Recovered - Amount in Sample}}{\text{Amount Added}} \times 100\%$$

13.1.3 Percent Relative Standard Deviation (%RSD)

$$\%\text{RSD} = \frac{\text{Standard Deviation}}{\text{Average CF}} \times 100$$

13.1.4 Percent Moisture

$$\% \text{ Moisture} = \frac{\text{g of Wet Sample - g of Dry Sample}}{\text{g of Wet Sample}} \times 100$$

13.1.5 Adjusted Estimated Practical Quantitation Limit for Samples

$$\text{Adjusted Estimated PQL} = \frac{(\text{PQL}) \times \text{Df}}{\text{D}}$$

where:

$$\text{D} = \frac{100 - \% \text{ Moisture}}{100}$$

DF = Dilution Factor

14.0 ACCEPTANCE OF DATA

14.1 Daily Calibration Check Standard (Required every 12 hour shift minimum)

Verification of the calibration curve with a single midlevel standard mix or multi-component calibration standard is obtained if the calculated concentration of all the compounds to be quantitated are (+/-) 15% of the expected value.

14.2 Breakdown Check (Required every 12 hour shift minimum)

Instrument Breakdown of DDT and Endrin is considered under control if the % Breakdown of each analyte is <= 15%.

14.3 Instrument Blank

The instrument blank is used to verify that the analytical system is free of contaminants. The instrument blank shall be free of any target compounds above half the quantitation limits and shall not contain any unusual interference. The instrument blank contains the same surrogates that are in all samples and standards.

14.4 Method Blanks

Method blanks are extracted with every batch of up to 20 samples to ensure that there is no contamination from the extraction process. The method blanks shall be free of any target compounds half of the quantitation limits and shall not contain any unusual interferences.

14.5 Matrix Spikes and Matrix Spike Duplicates

Matrix spikes and matrix spike duplicates are extracted with every batch of up to 20 samples to verify extraction efficiencies. Acceptance criteria are listed in Table 8.0.

14.5.1 MSB's are extracted with every batch of 20 samples as applicable to client requested protocol. Acceptance criteria are listed in Table 8.0

14.5.2 QC reference samples are extracted with every extraction batch of up to 20 samples to verify extraction efficiencies. Acceptance criteria are listed in Table 7.0.

15.0 REPORTING OF RESULTS

15.1 All results are reported to two significant figures. Water samples are reported in ug/L, soil samples are reported in ug/Kg dry weight and waste samples are reported in ug/Kg. Check reporting deliverables required from the traveller. All job packages require a case narrative and quality control approval report. The case narrative should outline in detail any problems with client samples during analysis. The following indicates the different levels of reporting.

Level I

- Case Narrative
- Sample Results

Level II

- Case Narrative
- Sample results
- Surrogate Recovery forms
- LCS & MS/MSD recovery forms

Level III/NJ
-Everything listed below, *Except* standard scans and area reports

CLP/NYSDEC

- Case Narrative
- Form 1 (Organic Analysis Data Sheet)
- Surrogate Recovery forms
- LCS & MS/MSD recovery forms
- MSB recovery form as applicable)
- Form 4C (Method Blank Summary)
- Initial Calibration Forms
- Analytical Sequence Form
- Breakdown Check Form
- Continuing Calibration Forms
- Form 10 (Pesticide/PCB Identification)
- Sample and Standard Scans and Area Reports
- Standard Concentration Summary
- GC/MS Confirmation (if applicable)

16.0 SUPPLEMENTAL DOCUMENTS

16.1 SOP for Pesticide Extract Sulfur Removal.

16.2 SOP for Pesticides in Water Extraction by Method 3510C.

16.3 SOP for Pesticides in Soil Extraction by Method 3550B.

16.4 Tables attached include the following:

Table 1.0 and 1.1 - Single-Component Calibration Concentrations
Table 2.0 - Multi-Component Concentration
Table 3.0 - Surrogate Mix
Table 3.1 - Surrogate Recovery Limits
Table 4.0 - Pesticide QC Check and Matrix Spike
Table 5.0 - Breakdown Standard Concentration

Table 6.0 - Practical Quantitation Limits (PQL)
Table 7.0 - QC Check Recovery Criteria
Table 8.0 - MS/MSD/MSB Recovery Criteria
Table 9.0 - TCLP Compounds and PQL's

17.0 POLLUTION PREVENTION

17.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

17.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

17.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

17.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

17.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

17.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

18.0 WASTE MANAGEMENT & POLLUTION PREVENTION

18.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention.

18.2 Personnel who handle or generate waste must be trained within six months of employment in proper waste handling and requirements.

18.3 Autosampler vials containing pesticides only shall be disposed of in the 5 gallon bucket labeled for Hexane waste.

19.0 REFERENCES

19.1 "Method 8000B - Determinative Chromatographic Separations", EPA SW846, 3rd Edition.

19.2 "Method 8081A - Organochlorine Pesticides by Gas Chromatography, SW846, 3rd Edition.

19.3 "Methods of Organic Chemical Analysis of Municipal and Industrial Wastewater", Federal Register Vol. 49, No. 209, October 26, 1984.

19.4 "USEPA CLP OLM03.2 Statement of Work" - pg.D-53/Pest, Section 10.1.8.2, Florisil Cleanup.

20.0 SUBSTANTIVE REVISIONS

20.1 Changed name of laboratory from AEN to STL; Feb. 8, 1999.

20.2 Revised section 8.3 changing standards hold time, revised section 9.6.2changing the calculation fo multiresponse pesticides. Revised section 9.7.3 to include reference to the Added Appendix A, revised section 10.4 changing the method blank criteria, revised section 13.5.2 to reference Table 7.0 for acceptance criteria; 03/05/99.

20.3 Added Terms and Definitions section, Pollution Prevention, and Waste Management; 2/15/2000. Added Initial calibration %RSD criteria and linear regression criteria. Corrected Mix 5 concentrations in Table 1.1 for alpha and gamma Chlordane.

20.4 Added to the Safety Section.

20.5 Added to the Safety section and Waste Management sections. Added EH&S Officer to Approval section. Removed Appendix A (multi-peak identification). Section 11.4.1 Added soxtherm blank information. Section 12.1 added autosampler. Fixed Isodrin and Chlorobenzilate reporting limits in Table 6.0. Changed Tables 3.1 and 7.0 to refer to LIM system for updated control limits. Section 12.3.2 added Soxtherm brief summary. Changed Toxaphene reporting limits in Table6.0-1/14/2004.

20.5 May 7, 2004- Added 40% Rule for reporting of results to Section 12.7.

20.6 January 14,2005-Added 4.3.2 Section concerning QC check solutions for Toxaphene and Technical Chlordane.

20.7 January 14, 2005 –Added 9.5.1 Section. Army Corp. requirements for low point calibration standard.

20.8 January 14, 2005-Added 9.6.1 Section. Army Corp. requirements for 3 levels of calibration for Multicomponent Pesticides.

20.9 January 14, 2005-Minutes added to 0.05 for retention time criteria in section 10.1.1.

20.10 January 14, 2005- Section 10.2.1 Added to calibration criteria further details.

20.11 January 14, 2005- Section 10.6.1 Linear regression calculation wording fixed.

20.12 January 14, 2005- Sections 11.6, 14.1, 14.2, and 14.3 Minor changes to wording.

20.13 January 14, 2005-Section 13.1.1 Absolute sign removed from calculation.

20.14 February 15, 2005- Sections 10.6.2 and 10.7.2 Guidance for Selecting Multi-component peaks.

20.15 February 15, 2005-Section 12.6.2 added.

20.16 February 15, 2005-Section 12.7.1 Clarified second column confirmation procedures.

20.17 February 15, 2005-Added to Section 11, Initial Demonstration and MDL criteria.

20.18 February 23, 2005 –Section 13.1.5, Changed naming to Adjusted Estimated PQL.

20.19 March 29, 2005 - Section 12.6.1, Changed PQL to MDL.

20.20 March 29, 2005 – Section 10.5.1 modified to analyze a CCV per 10 samples of poor matrix (not per 20). Section 10.5.2 Added for ACOE requirement of CCV per 10 samples.

20.21 April 19, 2005 – Sect 12.7.1 the Following statement was removed, "Data for more than two analyses shall not be submitted".

20.22 April 19, 2005 – Sect 19, added 8000C SW846 reference.

20.23 April 19, 2005 – Sect 11, clarified requirements for analysis of PT samples and also the use of control charts to establish in-house Control charts.

20.24 May 15, 2007 – Section 8.1 – Updated columns to those currently used. Sect. 9.3 Changed from 6months to 1 year. Sect. 9.4.1 – changed to one mix prepared with possibility of needing two in some circumstances. Section 9.5 – added lowest 6^{th} level of calibration curve as needed. Sect. 9.8 – Removed. Done in extraction dept. Sect. 10.2.1 – Coefficient is 0.990 or higher. Sect 10.3.1.2 – Reworded end ccal failures. Sect 11 – Renumbered entire section. Sect 11.10.1 – files now scanned, not filed. Sect. 11.9.2 removed filing system section. 11.10.3 – changed from Turbochrom to Target for runlogs. Sect 11.10.4 – changed CAR procedure to electronic. Sect 11.10.6 –

Removed reference to Labnet. Sect 11.11.2 – Changed from QCAR to Lims checklist for 1st/2nd level review. Sect 12.1 – updated to 6890's, columns, temperatures and ramps. Sect 12.9.1 changed to Chemstation and manually copied. Sect 12.9.3 – updated manual integration signing. Table 1.0 replaced with updated concentrations and levels.

20.25 9/12/2007 – Removed Sect 5.3 & 5.4 on cleanups. Sect 6.2 – footnote 1 removed. Sect 7.1 – added caps. Sect 8.1 – referenced 12.1. Added Tables 2.0-2.4 for preparation of stds. Sect 10.2.1 – added curve criteria for Linear regr. & quadratic. Sect 12.3.1 & 12.3.2 – removed methylene chloride for soils. Sect 12.6.2 – added ref to 8.1. Added sect 10.3.2 for second source analysis. Sect 10.1.1 – lowered min. RT window. Sect 11.10.4 – updated process.

Table 1.0
Compound Concentrations in Curve levels ug/ml

Pesticides	Level 0.5	Level 1	Level 2	Level 3	Level 4	Level 5
alpha-BHC	0.0025	0.005	0.01	0.025	0.05	0.10
beta-BHC	0.0025	0.005	0.01	0.025	0.05	0.10
delta-BHC	0.0025	0.005	0.01	0.025	0.05	0.10
gamma-BHC	0.0025	0.005	0.01	0.025	0.05	0.10
Heptachlor	0.0025	0.005	0.01	0.025	0.05	0.10
Aldrin	0.0025	0.005	0.01	0.025	0.05	0.10
Heptachlor Epoxide	0.0025	0.005	0.01	0.025	0.05	0.10
Endosulfan I	0.0025	0.005	0.01	0.025	0.05	0.10
Dieldrin	0.005	0.01	0.02	0.05	0.10	0.20
4,4'-DDE	0.005	0.01	0.02	0.05	0.10	0.20
Endrin	0.005	0.01	0.02	0.05	0.10	0.20
Endosulfan II	0.005	0.01	0.02	0.05	0.10	0.20
4,4'-DDD	0.005	0.01	0.02	0.05	0.10	0.20
Endosulfan Sulfate	0.005	0.01	0.02	0.05	0.10	0.20
4,4'-DDT	0.005	0.01	0.02	0.05	0.10	0.20
Methoxychlor	0.025	0.05	0.10	0.25	0.50	1.00
Endrin Aldehyde	0.005	0.01	0.02	0.05	0.10	0.20
Endrin Ketone	0.005	0.01	0.02	0.05	0.10	0.20
alpha-Chlordane	0.0025	0.005	0.01	0.025	0.05	0.10
gamma-Chlordane	0.0025	0.005	0.01	0.025	0.05	0.10
Toxaphene		0.20	0.50	1.00	2.00	4.00
Technical Chlordane		0.05	0.10	0.20	0.40	0.80
Tetrachloro-m-xylene (surrogate)	0.0025	0.005	0.01	0.025	0.05	0.10
Decachlorobiphenyl (surrogate)	0.005	0.01	0.02	0.05	0.10	0.20
* Isodrin	0.0025	0.005	0.01	0.025	0.05	0.10
* Chlorobenzilate	0.025	0.05	0.10	0.25	0.50	1.00
Mirex	0.0025	0.005	0.01	0.025	0.05	0.10
Alachlor	0.025	0.05	0.10	0.25	0.50	1.00

* Appendix 9 compounds

Table 2.0
Preparation of Pest Mix A/B

Pest Mix A/B

Prepared in
Iso-octane

Pest Mix A/B - Interm (level 5)	Initial Conc ug/ml	Amount Used ml	Final Volume mls	Final Conc. ug/ml	GINDABINT_
Mix A & B (8/16/80) (#32292)	8	2.5	200	0.10	
TCX	200	0.1	200	0.10	
DCB	200	0.2	200	0.20	

Pest Mix AB - Level 0.5	Initial Conc.	Amount Used	Final Volume	Final Conc.	made as needed
Pest A/B -Interm. LVL5	0.01	0.1	0.2	0.0025	
TCX				0.0025	
DCB				0.0050	

Pest Mix AB - Level 1	Initial Conc.	Amount Used	Final Volume	Final Conc.	GINDABWRK1_
Pest A/B -Interm. LVL5	0.10	2.5	50	0.005	
TCX				0.005	
DCB				0.010	

Pest Mix AB - Level 2	Initial Conc.	Amount Used	Final Volume	Final Conc.	GINDABWRK2_
Pest A/B -Interm. LVL5	0.10	5	50	0.010	
TCX				0.010	
DCB				0.020	

Pest Mix AB - Level 3	Initial Conc.	Amount Used	Final Volume	Final Conc.	GINDABWRK3_
Pest A/B -Interm. LVL5	0.10	50	200	0.025	
TCX				0.025	
DCB				0.050	

Pest Mix AB - Level 4	Initial Conc.	Amount Used	Final Volume	Final Conc.	GINDABWRK4_
Pest A/B -Interm. LVL5	0.10	25	50	0.050	

TCX	0.005
DCB	0.010

Table 2.1
Preparation of Miscelleanous compounds

AP9 Compounds

Prepare in Iso-octane

AP9 - Interm (level 5)	Initial Conc ug/ml	Amount Used **ul**	Final Volume mls	Final Conc. ug/ml	GINDABINT_
Isodrin	100	50	50	0.10	
Chlorobenzilate	1000	50	50	1.00	
Mirex	1000	5	50	0.10	

AP9 - Level 0.5	Initial Conc.	Amount Used	Final Volume	Final Conc.	
AP9 - Interm (level 5)	0.10	250	10	0.0025	Isodrin
	1.00			0.0250	Chlorobenzilate
	0.10			0.0025	Mirex

AP9 - Level 1	Initial Conc.	Amount Used	Final Volume	Final Conc.	GINDABWRK1_
AP9 - Interm (level 5)	0.10	500	10	0.0050	Isodrin
	1.00			0.0500	Chlorobenzilate
	0.10			0.0050	Mirex

AP9 - Level 2	Initial Conc.	Amount Used	Final Volume	Final Conc.	GINDABWRK2_
AP9 - Interm (level 5)	0.10	1000	10	0.0100	Isodrin
	1.00			0.1000	Chlorobenzilate
	0.10			0.0100	Mirex

AP9 - Level 3	Initial Conc.	Amount Used	Final Volume	Final Conc.	GINDABWRK3_
AP9 - Interm (level 5)	0.10	2500	10	0.0250	Isodrin
	1.00			0.2500	Chlorobenzilate
	0.10			0.0250	Mirex

AP9 - Level 4	Initial Conc.	Amount Used	Final Volume	Final Conc.	GINDABWRK4_

AP9 - Interm (level 5)	0.10	5000	10	0.0500	Isodrin
	1.00			0.5000	Chlorobenzilate
	0.10			0.0500	Mirex

Table 2.2
Preparation of Alachlor

Alachlor Curve

Prepared in Iso-Octane

Alachlor - (Intermediate)	Initial Conc ug/ml	Amount Used ml	Final Volume mls	Final Conc. ug/ml	GAlacINT_
Alachlor (Restek-32204)	1000	0.1	10	10	G_ALACHLOR_

Alachlor - (level 5)	Initial Conc ug/ml	Amount Used ml	Final Volume mls	Final Conc. ug/ml	**GAlacWRK5_**
Alachlor-Intermediate	10	1	10	1.0	GAlacINT_

Alachlor - Level 4	Initial Conc.	Amount Used	Final Volume	Final Conc.	**GAlacWRK4_**
Alachlor-Intermediate	10	0.5	10	0.500	GAlacINT_

Alachlor - Level 3	Initial Conc.	Amount Used	Final Volume	Final Conc.	**GAlacWRK3_**
Alachlor-Intermediate	10	0.25	10	0.250	GAlacINT_

Alachlor - Level 2	Initial Conc.	Amount Used	Final Volume	Final Conc.	**GAlacWRK2_**
Alachlor-Intermediate	10	0.1	10	0.100	GAlacINT_

Alachlor - Level 1	Initial Conc.	Amount Used	Final Volume	Final Conc.	**GAlacWRK1_**
Alachlor-Intermediate	10	0.05	10	0.050	GAlacINT_

Alachlor - Level 0.5	Initial Conc.	Amount Used	Final Volume	Final Conc.	made as needed
Alachlor-Intermediate	0.05	0.1	0.2	0.0250	GAlacINT_

Table 2.3
Preparation of Technical Chlordane

Technical Chlordane Prepare all standards in Iso-octane

Technical chlordane (Intermediate)	Initial Conc ug/ml	Amount Used ml	Final Volume mls	Final Conc. ug/ml	**G_TCLR_INT**
Technical chlordane (32021-Restek)	1000	0.5	10	50	G_TCLR_STK

Technical Chlordane - Level 1	Initial Conc.	Amount Used	Final Volume	Final Conc.	**G_TCLRLVL1**
TCLR -Intermediate	50	0.05	50	0.05	G_TCLR_INT
TCX/DCB Intermediate (5/10)	5	0.05	50	0.005	GSURINT_
Technical Chlordane - Level 2					**G_TCLRLVL2**
TCLR -Intermediate	50	0.1	50	0.10	G_TCLR_INT
TCX/DCB Intermediate (5/10)	5	0.1	50	0.01	GSURINT_
Technical Chlordane - Level 3					**G_TCLRLVL3**
TCLR -Intermediate	50	0.2	50	0.20	G_TCLR_INT
TCX/DCB Intermediate (5/10)	5	0.25	50	0.025	GSURINT_
Technical Chlordane - Level 4					**G_TCLRLVL4**
TCLR -Intermediate	50	0.4	50	0.40	G_TCLR_INT
TCX/DCB Intermediate (5/10)	5	0.5	50	0.05	GSURINT_
Technical Chlordane - Level 5					**G_TCLRLVL5**
TCLR -Intermediate	50	0.8	50	0.80	G_TCLR_INT
TCX/DCB Intermediate (5/10)	5	1	50	0.10	GSURINT_

Table 2.4
Preparation of Toxaphene

Toxaphene Prepare all standards in Iso-octane

Toxaphene (Intermediate)	Initial Conc ug/ml	Amount Used ml	Final Volume mls	Final Conc. ug/ml	G_TOX_INT
Toxaphene (32005-Restek)	1000	0.5	10	50	G_TOX_STK

Toxaphene - Level 1	Initial Conc.	Amount Used	Final Volume	Final Conc.	G_TOX_LVL1
Toxaphene -Intermediate	50	0.2	50	0.20	G_TOX_INT
TCX/DCB Intermediate (5/10)	5	0.05	50	0.005	GSURINT_
Toxaphene - Level 2					**G_TOX_LVL2**
Toxaphene -Intermediate	50	0.5	50	0.50	G_TOX_INT
TCX/DCB Intermediate (5/10)	5	0.1	50	0.01	GSURINT_
Toxaphene - Level 3					**G_TOX_LVL3**
Toxaphene -Intermediate	50	1	50	1.00	G_TOX_INT
TCX/DCB Intermediate (5/10)	5	0.25	50	0.025	GSURINT_
Toxaphene - Level 4					**G_TOX_LVL4**
Toxaphene -Intermediate	50	2	50	2.00	G_TOX_INT
TCX/DCB Intermediate (5/10)	5	0.5	50	0.05	GSURINT_
Toxaphene - Level 5					**G_TOX_LVL5**
Toxaphene -Intermediate	50	2	25	4.00	G_TOX_INT
TCX/DCB Intermediate (5/10)	5	0.5	25	0.10	GSURINT_

TABLE 3.0
SURROGATE MIX, UG/ML

Surrogate	Stock	Mix for Extractions (added to each sample and spike)
TCX	2.0	0.20
DCB	2.0	0.20

TABLE 3.1
SURROGATE RECOVERY LIMITS

Surrogate recovery limits are entered into the laboratory information system.

TABLE 4.0
PESTICIDE QC CHECK (LCS) AND FULL MATRIX SPIKE, UG/ML

Aldrin	2.0
Dieldrin	2.0
p,p'DDT	2.0
p,p'DDE	2.0
p,p'DDD	2.0
Endosulfan I	2.0
Endosulfan II	2.0
Endosulfan Sulfate	2.0
Endrin	2.0
Endrin Aldehyde	2.0
Heptachlor	2.0
Heptachlor Epoxide	2.0
alpha-BHC	2.0
beta-BHC	2.0
gamma-BHC	2.0
delta-BHC	2.0
Methoxychlor	2.0
Endrin Ketone	2.0
4,4'DDT	2.0

*See Table 7.0 for control limits

TABLE 5.0
BREAKDOWN STANDARD CONCENTRATION, UG/ML

Compound	Stock (Individual Compound)	Standard (Working)
Endrin	1.0	0.1
p,p'DDT	1.0	0.1

TABLE 6.0
PRACTICAL QUANTITATION LIMITS (PQL)

Analyte	Quantitation Limits, Water ug/L	Quantitation Limits, Soil ug/Kg
alpha-BHC	0.05	1.7
beta-BHC	0.05	1.7
delta-BHC	0.05	1.7
gamma-BHC	0.05	1.7
Heptachlor	0.05	1.7
Aldrin	0.05	1.7
Heptachlor Epoxide	0.05	1.7
Endosulfan I	0.05	1.7
Dieldrin	0.10	3.3
p,p'DDE	0.10	3.3
Endrin	0.10	3.3
Endosulfan II	0.10	3.3
p,p'DDD	0.10	3.3
Endosulfan Sulfate	0.10	3.3
p,p'DDT	0.10	3.3
Methoxychlor	0.50	17
Toxaphene	2.0	67
Technical Chlordane	0.50	17
Endrin Aldehyde	0.10	3.3

* Endrin Ketone	0.10	3.3

**Endrin Ketone is not on the Appendix IX list of reported compounds.

TABLE 6.0 - CONTINUED
PRACTICAL QUANTITATION LIMITS (PQL)

Analyte	Quantitation Limits, Water ug/L	Quantitation Limits, Soil ug/Kg
alpha chlordane	0.05	1.7
gamma chlordane	0.05	1.7
* Isodrin	0.05	1.7
* Chlorobenzilate	0.5	17

* App.IX Compounds

**Endrin Ketone is not on the Appendix IX list of reported compounds.

TABLE 7.0
QC CHECK CRITERIA

LCS control limits are entered into the Laboratory Information System.

TABLE 8.0
MS/MSD/MSB RECOVERY CRITERIA

MS/MSD/MSB control limits are entered into the Laboratory Information System.

TABLE 9.0
TCLP PESTICIDES

Analyte	Quantitation Limits, ug/L
Technical Chlordane	2.5
Toxaphene	12.5
Endrin	0.50
Heptachlor	0.25
Heptachlor Epoxide	0.25
Lindane	0.25
Methoxychlor	2.5

Figure 1.0

STL - Connecticut

GC SEMIVOLATILES INJECTION LOG Instr. hp6890-7.i

Standards Codes:	Routine Maintenance Performed:	Date: 06-APR-2007 14:22
	Cut & Cleaned: ( )	QC Batch: CD7354pest.b
	Changed Sleeve: ( )	
	Other: ( )	

Data File	Client ID	Sample ID	ALS	DF	Analyst	Reagent	Comments
D7354001.D	IBS	IBS	1	1	D.PASSARELLA	12118	
D7354002.D	IND0.5	IND0.5	2	1	D.PASSARELLA	22876	
D7354003.D	IND1	IND1	3	1	D.PASSARELLA	14777	
D7354004.D	IND2	IND2	4	1	D.PASSARELLA	22872	
D7354005.D	IND3	IND3	5	1	D.PASSARELLA	22874	
D7354006.D	IND4	IND4	6	1	D.PASSARELLA	28513	
D7354007.D	IND5	IND5	7	1	D.PASSARELLA	5208	
D7354008.D	TCLR1	TCLR1	8	1	D.PASSARELLA	12117	
D7354009.D	TCLR2	TCLR2	9	1	D.PASSARELLA	6325	
D7354010.D	TCLR3	TCLR3	10	1	D.PASSARELLA	12119	
D7354011.D	TCLR4	TCLR4	11	1	D.PASSARELLA	12122	
D7354012.D	TCLR5	TCLR5	12	1	D.PASSARELLA	12123	
D7354013.D	TOXAPH	TOXAPH	13	1	D.PASSARELLA	6326	
D7354014.D	ICV	ICV	14	1	D.PASSARELLA	19801	
D7354015.D	PIBLK	PIBLK	15	1	D.PASSARELLA	5722	
D7354016.D	MB 220-4870/1-AA	MB 220-4870_1-AA	16	1	D.PASSARELLA		
D7354017.D	LCS 220-4870/2-AA	LCS 220-4870_2-AA	17	1	D.PASSARELLA		
D7354018.D	MB 220-4558/1-AA	MB 220-4558_1-AA	18	1	D.PASSARELLA		
D7354019.D	LCS 220-4558/2-AA	LCS 220-4558_2-AA	19	1	D.PASSARELLA		
D7354020.D	EAST HAMP	220-1093-A-1-C	20	1	D.PASSARELLA		
D7354021.D	GLAST	220-1093-B-2-D	21	1	D.PASSARELLA		
D7354022.D	SP-332	220-1266-A-1-A	22	20	D.PASSARELLA		
D7354023.D	SP-332 MS	220-1266-A-1-B MS	23	20	D.PASSARELLA		
D7354024.D	SP-332 MSD	220-1266-A-1-C MSD	24	20	D.PASSARELLA		
D7354025.D	SP-333	220-1266-A-2-A	25	40	D.PASSARELLA		
D7354026.D	SP-334	220-1266-A-3-A	26	20	D.PASSARELLA		

Witnessed By: [signature] Date: 4/10/07 Page No. _____

Figure 2.0

May, Dawn

Subject: 220-1461 Roux 05/15/07
Start Date: Tuesday, May 15, 2007
Due Date: Wednesday, May 16, 2007

Status: In Progress
Percent Complete: 0%

Total Work: 0 hours
Actual Work: 0 hours

Owner: Maturo, Kim

SEVERN TRENT STL

NON-CONFORMANCE/CORRECTIVE ACTION FORM

A. Originator Information **Client Inquiry:**

Client:	*Roux*	*Job Number:* *Sample number(s):*	*220-1461* *#4 TCLP*
Date/time Initiated:	*5/15/07*	*Fraction:*	*TCLP Pest*
Lab/Client Originator:	*Kim Maturo*	*Date/time response due:*	*5/16/07*

Groups Involved:

	Sample control		Wet Chemistry		Metals	X	Organic Extractions
X	Gas Chromatography		MS-VOA		MS-SV		Report Generation
X	Client Services		Subcontractor				EDD

Detailed Description of Potential Problem:
Both surrogates failed VERY low in the sample and it therefore requires reextraction as soon as possible since the job is late. Sarah-Please let Dan know ASAP if there isn't sufficient leachate. 5/15/07 KM

B. Quality Assurance Information
Recommended Corrective Action:

C. Final Resolution
Describe What Happened and Corrective Action Taken:

Supervisor Review: Date: Date/time of Client notification:

Date EDD/Report Revision Sent:_____

D. Quality Assurance Final Approval (QA Manager or designee use only)

Corrective Action Approved:_____

STL Doc.# QAF00204.CT

Figure 3.0

STL Connecticut

LIMS Organics Data Review Checklist

Batched/First Level	Second Level
Prep Batch created (VOA) -TCLP's/MeOH Preps	
Check Method	
Dilutions appropriate	
Q-Editing checked	
TIC's called	Recheck
AD Worksheet (VOA) Init/FV	Recheck
AD Reagents (VOA) codes/amts	Recheck
Sample Results -RL's look correct -Flagging correct -RE/DL suffixes as needed	Recheck
GC Dual Column criteria applied -P/A sample results -P/S surrogates	Recheck
Surrogate recoveries present/flagged	Recheck
TIC's reported correctly	Recheck
QC linking correct	Recheck
NCM's created (record batch#)	Read

JOB LOCKED (PM Desktop)

Doc# QAF04300.CT

GAS CHROMATOGRAPHY INSTRUMENT MAINTENANCE LOG

Instrument ID ____

Date	*Analyst*	*Problem*	*Maintenance Performed*

Instrument ID ____

Date	*Analyst*	*Problem*	*Maintenance Performed*

Instrument ID ____

Date	*Analyst*	*Problem*	*Maintenance Performed*

Instrument ID ____

Date	*Analyst*	*Problem*	*Maintenance Performed*

FIGURE 4.0

Title: SOP for GC Method 8082 PCBs
[Method SW846 8082]

Approvals (Signature/Date):

Technical Manager	9/27/07 Date	Health & Safety Manager / Coordinator	10-15-07 Date
Quality Assurance Manager	10/17/07 Date	Laboratory Director	10/19/07 Date

This SOP was previously identified as SOP No. GCS02308.CT.

Any reference within this document to Severn Trent Laboratories, Inc. or STL, should be understood to refer to TestAmerica Laboratories, Inc. (formerly known as Severn Trent Laboratories, Inc.)

Facility Distribution No. _____ Distributed To:_____

1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 This method defines the specific steps for analyzing and determining the concentration of various polychlorinated biphenyls (PCBs) in multimedia, multi-concentration samples.

2.2 It is the policy of STL and of the chromatography group to ensure that we administer contracts and orders for goods and services in a manner that is fully compliant with governmental laws and regulations, as well as the TestAmerica Policy Statement on Business, Ethics and Conductivity.

2.3 Refer to Table 5 of this SOP for the list of parameters.

2.3 The document control number for this SOP is CT-GCS-23, Rev 8.

3.0 TERMS AND DEFINITIONS

3.1 There are many definitions used with in the laboratory, which may be generic to all laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used with in the laboratory, reference the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 This method outlines the gas chromatographic procedure for the detection of polychlorinated biphenyls. Samples are extracted using the proper extraction technique. The extracts are analyzed by gas chromatography with an electron capture detector.

4.2 This SOP is based on the following methods:

- EPA Method 8082 (PCB's as Aroclors by Gas Chromatography)
- EPA Method 8000B (Determinative Chromatographic Separation)
- EPA Method 8000C (Determinative Chromatographic Separation)

4.3 Deviations to Method

4.3.1 The laboratory at this time, does not analyze samples for individual PCB congeners.

5.0 INTERFERENCES

5.1 Phthalate esters can interfere with PCB determinations; avoid any contact with plastics to best minimize this problem.

5.2 Sulfur is also an interference; this can be removed by performing sulfur clean-up on the extract by using either Copper or Mercury. To use copper, take 2ml of sample extract and add 4grams of Copper beads. Shake for 1minute, then let sit for 10minutes. Pipette off extract and analyze. Do not let sit for longer than 10minutes or the loss of compounds is possible. When using mercury, refer to the Pesticide Extract Sulfur Removal SOP.

5.3 If an extract is to be analyzed for PCB's only, then an optional sulfuric acid cleanup may be performed. This cleanup procedure will remove contamination, which may interfere with the analysis of the eluting PCB's, however; the procedure may also cause degradation of other pesticide and surrogate compounds. If contamination, due to sample matrix, is suspected in the sample, or if the original extract analysis exhibits interferences, then the extract may be cleaned up with concentrated sulfuric acid and reanalyzed. A portion of the original method blank is also acid cleaned up and analyzed.

5.3.1 Acid cleanup is performed by adding one part sulfuric acid to two parts of the hexane extract in a clean vial. Tighten the vial and agitate the sample for thirty seconds. After cleanup, pipet the extract layer (top layer) to another vial. Repeat the cleanup procedure until the acid layer is light brown to orange in color. Complete the acid cleanup procedure by performing a sulfur cleanup. This cleanup must also be performed on the corresponding method blank and QC if all samples required cleanup, or a portion of the method blank and QC if only some of the samples required cleanup. Refer to Pesticide Extract Sulfur Removal SOP. Document in the case narrative each sample on which acid and/or sulfur cleanup was performed.

6.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

6.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

6.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

7.1 Sample Containers

- Water samples are collected in 2x1 liter amber glass containers with Teflon-coated liner and teflon coated caps..
- Soil samples are collected in 250 or 500 mL glass containers with Teflon coated caps.
- Sample bottles are never reused.

7.2 Sample Collection

- Samples are secured against breakage in the shipping containers and kept at 4°C for transport to the laboratory. Samples should arrive at the laboratory the next day following collection.

7.3 Sample Preservation

- Samples are preserved by cooling to 4°C.

7.4 Holding Times

- Water samples must be extracted within seven days from collection.
- Soil samples must be extracted within 14 days from collection.
- Aqueous samples requiring NYSDEC ASP must be extracted within five days from VTSR.
- Soil samples requiring NYSDEC ASP should be extracted within 14 days from collection.
- All extracts must be analyzed within 40 days from date of extraction (40 days from collection for NYSDEC ASP).

8.0 APPARATUS AND MATERIALS

8.1 Sample extracts are analyzed on a gas chromatograph (GC) equipped with an electron-capture detector (ECD), autosampler, data collection system and all other required accessories. The following columns are acceptable for analysis:

See section 12.1 for instrumentation specifications

- Restek Rtx-CLPesticides 15 meter
  0.53mm ID 0.50um film thickness
  or equivalent

- Restek Rtx-CLPesticidesII 15meter
  0.53mm ID 0.50um film thickness
  or equivalent

- Turbochrom Data Acquisition Software System or Chemstation software

- Target Software

- Various sizes of syringes, volumetric pipets, volumetric flasks, pipet bulbs

- 0.8 ml and 1.8 ml autosampler vials and caps.

- Borosilicate glass transfer pipets/transfer bulbs.

- Safety glasses, non-powdered polyvinyl gloves, fume hood.

- Properly cooled refrigerators each for sample and standard storage.

- Standard and Instrument Maintenance logbooks.

- Sample Injection logbooks.

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Solvents: Hexane, Acetone, Toluene and Isooctane (2,2,4-trimethylpentane) should be pesticide grade or equivalent.

9.2 Commercially prepared stock standards can be used if they are certified and pretested by the manufacturer.

9.3 All stock and working standards are stored in amber screw top bottles at 4°C and replaced after 1 year or earlier if routine QC tests indicate a problem.

9.4 Calibration Stock Standard

9.4.1 **Multi-component Standards** - A five point calibration curve for combination standard mix of Aroclor-1016 and Aroclor-1260 is performed and a single midpoint calibration

standard of all other Multicomponents. All of these standards are diluted from individual stocks. For Multi-component Stock and Standard concentrations see Table 1.0. For preparation of standards see Table 2.0.

9.5 **Surrogate Standard** is prepared to monitor the performance of the extraction and analytical system. Samples, standards and blanks are spiked with pesticide surrogates. Two surrogates, 2,4,5,6-tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB), are spiked with the proper amount at a level of 0.2 ug/L for waters and soils are spiked at a level of 6.7 ug/Kg. (See Table 3.0).

9.6 **Quality Control Check and Matrix Spiking Standard Solutions**: A mixture of Aroclor 1016/1260 is used as the LCS spike solution. A mixture of Aroclor 1016/1260 is also used as the MS/MSD spike solution. 1.0 ml is spiked into each aliquot of the sample for the matrix spike and matrix spike duplicate, and to 1.0 L of reagent water for the QC check sample. (See Table 4.0).

10.0 CALIBRATION

10.1 RT Windows are established by making three injections of the mid concentration standard throughout the course of a 72-hour period and calculating 3 x ± the standard deviation. Retention time windows shall be calculated for each compound on each GC column whenever a new column is installed.

10.1.1 RT windows are established as described above, however, the laboratory has established a minimum RT window of 0.05.

10.2 Calibration Standards

Initial calibration standards are analyzed by injecting 1 ul of five levels of AR1660 standard, and one level of all other multi-response PCB's. (Aroclor 1016 and 1260 include all congeners present in the different regulated Aroclors).

10.2.1 The initial calibration criteria is 20% RSD. If the %RSD cannot be met using the average calibration factor, an alternate curve type can be used. The criteria are 0.990 or higher for the coefficient.

10.2.2 In determining the best fit of a curve, using either linear regression or Quadratic, the analyst shall choose the best fit possible based on both the coefficient and the slope of the line (b). The coefficient should be greater than 0.990 and as close to 1 as possible. The slope of the line (b) should be as low a value as possible. The analyst may choose between linear regression, a weighted linear regression or a Quadratic fit. The line may be forced through zero, if the (b) value by other means, would result in possible negative results.

10.3 The working calibration range of this method is defined by the initial calibration curve. All

extracts with target compounds exceeding the curve must be diluted to within the upper half of the calibration range.

10.3.1 The daily calibration verification is acceptable if all of the following are true.

10.3.1.1 The calibration verification standard must have all compounds (+/-) 15% difference of their expected value. Because of the low concentration of pesticide standards injected on a GC/EC, column adsorption may be a problem when the GC has not been operated for a day. Therefore, the GC column should be primed by injecting a standard mixture approximately 20 times more concentrated than the midlevel standard.

If the calibration verification standard fails, then it can be rerun. A new calibration curve will be run, if warranted, due to repeated failure. When the calibration verification standard fails, all sample that were injected after the last standard that met the QC criteria must be evaluated to prevent any mis-quants and possible false negatives. Depending on the compound failures the extracts may need reinjection. More frequent analysis of standards will minimize the number of reruns for QC failures.

10.3.1.2 An instrument blank standard is run after the calibration verifications standards and prior to any samples to ensure the instrument and its autosampler are clean.

10.3.2 All initial calibrations must be verified with a standard obtained from a second source manufacturere or lot. The same manufacturer may be used, if the lot can be demonstrated from the manufacturer as prepared independently from other lots. Traceability shall be to a national standard when commercially available. The % difference of this standard should pass within +/- 25% from the initial calibration curve. If the second source standard fails to meet criteria, check the preparation of both the initial curve and the second source standard for errors. It may be necessary to re-prepare a solution to check.

10.4 When analyzing samples, any extract that contains a target compound that exceeds the high level calibration must be diluted to within the calibration range.

10.5 Calibration Verification - (Each 12 hour shift)

10.5.1 A single multi-compound standard, is analyzed at a minimum of every 20 samples and at the end of the analysis sequence (It is recommended every 10). The % difference of the average of the 5 peaks for an aroclor, must not exceed a 15 percent. When this criteria is exceeded, inspect the GC system to determine the cause and perform whatever maintenance is required before recalibrating and proceeding with sample analysis.

10.6 Calculations for water samples:

10.6.1 An average calibration factor is calculated for each peak (using 3-5 peaks) and surrogate

from the initial calibration.

Multicomponent Calculation

$$ug/L = \frac{(X)\ \text{Final Volume of extract (uL)} \times \text{Dilution Factor}}{(Y)\ \text{Sample Volume extracted (mLs)} \times \text{ul inj.}}$$

X = The area of one of the 3-5 peaks from sample.
Y = The average CF for the corresponding peak from standard.

A Cf must be calculated for 3-5 peaks. The final concentration reported is calculated using the average of the final result for each of the 3-5 peaks.

10.7 Calculations for soil and oil samples:

10.7.1 An average calibration factor is calculated for each peak (using 3-5 peaks) and surrogate from the initial calibration.

Multicomponent Calculation

$$ug/Kg = \frac{(X)\ \text{Final Volume of Extract (uL)} \times \text{Dilution Factor}}{(Y)\ \text{Sample Volume Extracted(g)} \times \text{Decimal \% Solids} \times \text{ul inj.}}$$

X = The area of one of the 3-5 peaks from sample.
Y = The average CF for the corresponding peak from standard.

A Cf must be calculated for 3-5 peaks. The final concentration reported is calculated using the average of the final result for each of the 3-5 peaks.

Note: Oils will not have a decimal percent solid.

10.7.2 When more than one multi-component is detected in a sample, non-overlapping peaks are chosen for quantitation. If it is not possible to choose non-overlapping peaks, peaks with the least amount of overlap are chosen. Refer to Appendix A for the most common peaks chosen for quantitation. Three to five of the peaks are used to calculate the calibration curve, and any results from corresponding samples.

10.8 Corrective Action for Initial Calibration

10.8.1 If the technical acceptance criteria for the initial calibration are not met, inspect the system for problems. It may be necessary to change the column, bake out the detector, clean the injection port, or take other corrective actions to achieve the acceptance criteria.

10.8.2 Corrective Action for Calibration Verification

10.8.3 If the technical acceptance criteria for the calibration verification are not met, inspect the system for problems and take corrective actions to achieve the criteria.

10.8.3 Major corrective actions such as replacing the column or detector will require a new initial calibration. A return to control indicating the next passing curve shall be documented in the maintenance log.

10.8.4 Minor corrective actions may not require a new initial calibration provided a calibration check meets all acceptance criteria.

11.0 QUALITY CONTROL

11.1 Refer to Table 5.0 for Practical Quantitation Limits (PQL's) for all compounds.

11.2 Matrix spike (MS), matrix spike duplicates (MSDs) and matrix spike blanks (MSB's), if applicable, are analyzed within every set of 20 samples or less. Recoveries must be within the limits listed in Table 4.1 of this SOP. If these criteria are not met, but the blank spike data meet all of the recovery criteria, then the MS/MSD are documented as having matrix interferences. If the blank spike fails, check for instrument and/or column related problems and reanalyze the spikes. If the problem is corrected, the samples are reanalyzed.

11.3 A QC check sample is performed at a frequency of one per 20 samples extracted of similar matrix. See Table 4.0 for spike compounds and control limits.

11.4 Blanks

11.4.1 Method Blank

Method blanks are spiked with surrogates, extracted, and analyzed following the same procedure that is used with the associated samples. A water method blank is one liter of reagent water and a soil method blank is 30 grams of sodium sulfate for sonication extraction and 15 grams of sodium sulfate for automated soxtherm extraction.

Frequency - method blanks must be analyzed with each case, 20 samples of a similar matrix, or whenever samples are extracted by the same procedure, whichever is more frequent.

Acceptance criteria - method blanks must contain less than half the PQL for all the target compounds listed in Table 1.0 & 1.1.

All samples associated with an unacceptable method blank must be re-extracted and reanalyzed.

11.5 Surrogates

11.5.1 The surrogates TCX and DCB are added to each standard, sample, blank and QC prior to extraction. Results for the surrogates are calculated from the Pestcide INDA Mixes if Pesticides are requested. If the sample is for PCB only, then the surrogates can be calculated using the Aroclor 1016/1260 calibration standards if the INDA mixes were not analyzed.

11.5.2 The QC limits for surrogate recovery are listed in Table 3.1 and pertain to all samples, blanks and spikes.

11.6 Daily Calibration Verification: One per 12 hour shift

Instrument Blank:	One per 12 hour shift
MS/MSD:	Every 20 samples
QC Check:	Every extraction batch
Surrogate:	All samples and standards
Method Blank:	One per batch

11.7 Analytical Documentation Procedures

11.7.1 Instrument Batches

An instrument batch is created for each analytical sequence to organize all the associated data. Batch designations are of the format:

XXnnn

where: XX = instrument identifier
nn = number of batch

(i.e. C4001)

Instrument batches are number sequentially so a unique batch identifier identifies each analytical sequence. The batch consists of a file folder with all the associated QC information for the analytical sequence. The raw data is then scanned for all initial and continuing calibratons.

11.7.2 Data Archiving

All data files are archived on a daily basis using a 12.0gb data storage cartridge. The associated method files are also archived daily to provide an accurate historical record. Care shall be exercised when purging data off the hard drives to ensure that all data being purged has been archived.

11.7.3 Instrument Run Logs

It is STL's policy that all measurement data be recorded in logbooks or on preprinted log sheets in permanent ink. Transcriptions shall be avoided whenever possible. The record shall reflect the measurement performed and all appropriate details for conclusions related to the measurement. The record shall be signed and dated by the individual performing the measurement on the day the measurement is performed. Corrections shall be made by drawing a single line through the error, and initialing and dating the correction. A secondary authorization of the logbook is required and shall be performed by the department's manager or designee.

Each instrument has its own set of bound run logs (see Figure 1.0) which are sequentially numbered and paginated. Run logs are filed in the laboratory once they have been filled, for future reference. Each analytical sequence shall be started on a new page of the log and continued on the next page, if necessary. The header information designating the standard codes used shall be completed for each sequence. All standards used are recorded in this field for future traceability. The data file, job number, sample number, quantitation factor, dilution factor, analyst's signature, and date are recorded.

11.8.5 Corrective Action Reports

A corrective action report (CAR) is initiated when a problem is encountered during analysis, data reduction or deliverables preparation, data validation, or when any deviations from this SOP occur. The CAR is initiated by the analyst or dept. manager first identifying the problem through the NCM module in the LIMS system. It is then electronically forwarded to all appropriate departments, QA officer, and Lab Manager. Reference SOP for correction action reports.

11.8.6 Chain of Custody Record

When samples are removed from storage for preparation or analysis they must be signed out utilizing the chain of custody record (COC). The samples shall then be signed back in on the COC upon their return to storage or designated "used" if the sample volume is consumed during the preparation or analysis.

11.8.7 Sample Tracking Record

The Sample Control department does notification of sample arrival by issuing a preliminary notification sheet. Samples are tracked for extraction and analysis by using the laboratory's LIMS system.

11.9 Quality Control Check Points

11.9.1 Analysis quality control approval report

Specific quality control checkpoints have been established for the analysis of sample extracts. The specific check points in the analysis logbook, are initialed and dated by the analyst to ensure the consistency and accuracy of the data produced.

11.9.2 Specific quality control checkpoints have been established for the preparation of data deliverables, which are monitored through a Lims Organics Data Review Checklist – Doc # QAF04300.CT. The specific check points must be reviewed by the analyst 1st level reviewing and the secondary reviewer 2nd level revieweing the data to ensure the consistency and accuracy of the data produced. Refer to Figure 3.0 for the document and specific control points covered.

12.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

12.1 HP5890 Series II GC's with dual ECD's or HP6890's with Micro ECD's and 7673A Twin Tower Autosampler or CTC (Leap Technologies) Autosampler is currently being used to run Pesticides/PCBs under this SOP. Instruments, columns and guidances conditions are as follows:

a) Column flow: ~8 mL/min He
Detector Make-up Flow: 65° mL/min N_2
Injector Temperature: Tracks oven temperature +3 deg. C
Detector Temperature: 330°C

Temperature Programming:
Initial Temperature: 150°C Hold 0.8 min
Initial Ramp: 11°/min to 208, 0.6min Hold
Final Ramp and Temp.: 35°/min to 315, Hold 0.7 min

Instrument and column conditions may have equivalent programming as long as all method QC requirements are met.

12.2 Aqueous Sample Preparation for PCBs:

12.2.1 Brief Summary:
A 1 liter sample aliquot is spiked with the surrogate and extracted with methylene chloride at a pH between 5 and 9. Using either the separatory funnel extraction method or the continuous liquid-liquid extraction method is acceptable. The methylene chloride extract is dried and solvent exchanged to hexane and adjusted to a final volume of 10 mL.

A 1 uL aliquot of the sample extract is injected into the gas chromatograph (GC). The GC separates the compounds. Compound identification is performed by the comparison of GC

retention times to those of known analytical standards. Quantitative analysis is performed by the comparison of compound peak height or peak areas to those of an analytical standard at a known concentration.

12.2.2 For TCLP samples, 800 mLs of reagent water is added to 200 mLs of the TCLP leached sample and the procedure would follow Section 12.2.1.

12.2.3 Sample Extraction

Refer to STL Standard Operating Procedure water extraction.

12.3 Soil Sample Preparation for PCB's

12.3.1 Brief Summary

A 30 g sample aliquot is spiked with surrogate and extracted with a 1:1 mixture of hexane/ acetone using the sonication extraction method. The hexane /acetone extract is dried and concentrated to a final volume of 10 mL. A 1 uL aliquot of the sample extract is injected into the gas chromatograph (GC). The GC separates the compounds. Compound identification is performed by the comparison of GC retention times to those of known analytical standards. Quantitative analysis is performed by the comparison of compound peak height or peak areas to those of an analytical standard at a known concentration.

12.3.2 A 15 g sample aliquot is spiked with surrogate and extracted with a 1:1 mixture of hexane/ acetone using the soxtherm apparatus. The hexane /acetone extract is dried and concentrated to a final volume of 5 mL. The sample extraction must be completed within 14 days of sample collection. A 1uL aliquot of the sample extract is injected into the gas chromatograph (GC). The GC separates the compounds. Compound identification is performed by the comparison of GC retention times to those of known analytical standards. Quantitative analysis is performed by the comparison of compound peak height or peak areas to those of an analytical standard at a known concentration.

12.3.3 Sample Extraction

Refer to STL Standard Operating Procedure for soil extraction by sonication and soxtherm.

12.4 Sample Analysis Procedures

12.4.1 Sample Extract Analysis

Sample extracts are removed from storage in the G.C. instrument room and are signed out on the extract chain of custody form. All sample extracts are signed back in after they are returned to storage.

Make sure all instrumental operating conditions are correctly set.

In a vial with a 200 ul insert, load a 200 ul aliquot of sample extract. A 1.0 ul injection of the sample extract onto the GC column is made with an autosampler and then the GC temperature program sequence is started.

This method is intended to achieve the quantitation limits whenever possible. If sample chromatograms have interfering peaks, high baseline, or off-scale peaks, then those samples must be reanalyzed following dilution, cleanup, or reextraction. No limit is placed on the number of reextractions of samples that may be required because of contaminated method blanks.

The sample must be analyzed at the most concentrated level that is consistent with achieving satisfactory chromatography (defined in Section 12.7).

No target analyte concentrations may exceed the upper limit of the initial calibration.

12.5 Qualitative Analysis

12.5.1 Target Compounds

The identification is based on retention time data as well as Aroclor pattern.

If the pattern appears to be that of an Aroclor, and is greater than the PQL, that sample may be confirmed by analysis on a second column of dissimilar phase, see section 8.1.

12.6 Quantitative Analysis

12.6.1 Target Compounds

Target compounds are quantitated by the external standard technique using peak area and the calibration factor determined during the initial calibration sequence.

When compound concentrations are below the PQL, but the compound meets identification criteria, report the concentration with a "J" qualifier.

When a compound exceeds the linear working range of the initial calibration, the sample must be diluted to bring the analyte concentration within the calibration range.

The following are guidelines on performing dilutions and exceptions to this requirement:

- If the response is still above the high calibration point after the dilution of 1:100,000, the laboratory shall contact the client.

- Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range.

12.6.2 The lab routinely reports the higher concentration of the results between the two columns used for analysis. If there is a greater than 40% difference between the two columns, the lower result is reported.

12.7 Instrument Maintenance

12.7.1 Preventative Maintenance

All instrumentation is covered by a service contract with an external instrumentation service vendor, or by STL personnel trained in preventative maintenance. All instrument preventative maintenance is performed according the manufacturers recommended procedures, by trained personnel. All preventative maintenance shall be thoroughly documented in the maintenance log (see Figure 4.0), as to a description of the maintenance performed, the date performed, and the personnel performing the maintenance.

12.7.2 Corrective Maintenance Determinants and Procedures

Corrective maintenance is deemed necessary when the analytical system, after reanalysis, cannot meet calibration, resolution, chromatography, breakdown, or other protocol specific QC criteria. Corrective maintenance may include, but is not limited to, decontamination of the system, injection port cleaning, column cutting or replacement, syringe cleaning or replacement, or detector baking out or replacement. All corrective maintenance is performed according the manufacturers recommended procedures, by trained personnel. All corrective maintenance shall be thoroughly documented in the maintenance log, as to a description of the maintenance performed, the date performed, and the personnel performing the maintenance. A return to control documenting the next valid curve shall be noted in the maintenance log in the same section.

12.7.3 Maintenance Authorization

The department's manager, or designee authorizes all preventative and corrective maintenance. When maintenance is deemed necessary, a service call is placed for all equipment covered under a service contract, by the department's manager, or designee.

12.8 Data System

12.8.1 Data Acquisition and System Operation

Data is acquired from sample analyses using the Perkin Elmer TurboChrom or Chemstation computer system. Analytical batches are set up with all the associated

sample ID, dilution, and data file information. Automated post-acquisition is qued with the appropriate method file and sent to the Target data system for integration and quantitation. Turbochrom and/or Chemstation has instrument control.

12.8.2 Instrument Errors

System errors are logged to the system console at time of occurrence. The system manager shall be responsible for checking and providing corrective actions for all system errors.

12.8.3 Manual Integration Editing Flags

Manual integrations shall be performed when the automated integration does not yield correctly integrated baselines. Manual integrations are flagged by the data system with the "M" qualifier beside the concentration of any manually integrated compound. The analyst name is printed on the quantitation report. If a name does not appear on the report, the analyst must manually date and sign the page.

13.0 CALCULATIONS

See Sections 10.5.1, 10.5.2, 10.6.1 and 10.6.2 of this document.

13.1 Calculation of Calibration Factor

$$CF = \frac{\text{Peak Area of the Standard}}{\text{Mass Injected (ng)}}$$

13.1.1 Calculation of Percent Difference

The following formula is used to calculate % difference in the calculated versus expected values of standards.

$$\% \text{ Difference} = \frac{|\text{Calculated conc. - expected conc.}|}{\text{Expected conc.}} \times 100$$

13.1.2 Calculation of Surrogate, Spike and QC Check Recoveries

The following calculation is used for spiked sample recoveries.

$$\% \text{ Recoveries} = \frac{\text{Amount Recovered - Amount in Sample}}{\text{Amount Added}} \times 100\%$$

13.1.3 Percent Relative Standard Deviation (%RSD)

$$\%RSD = \frac{\text{Standard Deviation}}{\text{Average CF}} \times 100$$

13.1.4 Percent Moisture

$$\% \text{ Moisture} = \frac{\text{g of Wet Sample - g of Dry Sample}}{\text{g of Wet Sample}} \times 100$$

13.1.5 Adjusted Practical Quantitation Limit for Samples

$$\text{Adjusted PQL} = \frac{(\text{PQL}) \times \text{Df}}{D}$$

where:

$$D = \frac{100 - \% \text{ Moisture}}{100}$$

DF = Dilution Factor

14.0 ACCEPTANCE OF DATA

14.1 Daily Calibration Check Standard (Required every 12 hour shift)

Verification of the calibration curve with a multi-component calibration standard is obtained if the calculated concentration of all the compounds to be quantitated are (+/-) 15% of the expected value.

14.2 Instrument Blank

The instrument blank is used to verify that the analytical system is free of contaminants. The instrument blank shall be free of any target compounds above half the quantitation limits and shall not contain any unusual interferences.

14.3 Method Blanks

Method blanks are extracted with every batch of up to 20 samples to ensure that there is no contamination from the extraction process. The method blanks shall be free of any target compounds above half the quantitation limits and shall not contain any unusual interferences.

14.4 Matrix Spikes and Matrix Spike Duplicates

Matrix spikes and matrix spike duplicates are extracted with every batch of up to 20 samples to verify extraction efficiencies. Acceptance criteria is listed in Table 4.0.

14.4.1 MSB's are extracted with every batch of 20 samples as applicable to client requested protocol. Acceptance criteria are listed in Table 4.0

14.4.2 QC reference samples are extracted with every extraction batch of up to 20 samples to verify extraction efficiencies.

15.0 REPORTING OF RESULTS

15.1 All results are reported to two significant figures. Water samples are reported in ug/L, soil samples are reported in ug/Kg dry weight and waste samples are reported in ug/Kg.

Check reporting deliverables required from LIMS. All job packages require a case narrative and quality control approval report. The case narrative should outline in detail any problems with client samples during analysis. The following indicates the different levels of reporting.

Level I

- Case Narrative
- Sample results

Level II

- Case Narrative
- Sample Results
- Surrogate Recovery form
- LCS and MS/MSD recovery forms

Level III/NJ

-Includes everything listed below, *Except* standard scans and area reports.

NJ/CLP/NYSDEC

- Case Narrative
- Form 1 (Organic Analysis Data Sheet)
- Surrogate Recovery form
- LCS and MS/MSD recovery forms
- MSB recovery form as applicable
- Form 4C (Method Blank Summary)

- Initial Calibration Forms
- Analytical Sequence Form
- Breakdown Check Form
- Continuing Calibration Forms
- Form 10 (Pesticide/PCB Identification)
- Sample and Standard Scans and Area Reports
- Standard Concentration Summary
- GC/MS Confirmation (if applicable)

16.0 SUPPLEMENTAL DOCUMENTS

16.1 SOP for Pesticide Extract Sulfur Removal.

16.2 SOP for Pesticides in Water Extraction by Method 3510C.

16.3 SOP for Pesticides in Soil Extraction by Method 3550B.

16.4 Tables attached include the following:

Table 1.0 - Surrogate Calibration Concentrations
Table 2.0 and 2.1 - Multi-Component Concentration
Table 3.0 - Surrogate Mix
Table 3.1 - Surrogate Recovery Limits
Table 4.0 - Aroclor QC Information
Table 4.1 - Aroclor Matrix Spike Information
Table 5.0 - Practical Quantitation Limits (PQL)

17.0 POLLUTION PREVENTION

17.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

17.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

17.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

17.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

17.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

17.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

18.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention.

18.1 All autosampler vials containing PCB's should be disposed of in the 5 gallon bucket labeled for Hexane waste.

19.0 REFERENCES

19.1 "Method 8000C – "Gas Chromatography", EPA SW846, 3rd Revision.

19.2 "Method 8000B – "Gas Chromatography", EPA SW846, 3rd Edition
Project Dependent.

19.3 "Method 8082 - PCBs as Aroclors by Gas Chromatography, EPA SW846, 3rd Edition

19.4 "Methods of Organic Chemical Analysis of Municipal and Industrial Wastewater", Federal Register Vol. 49, No. 209, October 26, 1984.

19.5 "USEPA CLP OLM03.1 Statement of Work" - pg.D-53/Pest, Section 10.1.8.2, Florisil Cleanup.

20.0 SUBSTANTIVE REVISIONS

20.1 Revised section 8.3 changing the standard storage time frame, revised section 9.7.2 adding a reference to the added Appendix A - Chromatograms of Aroclors, revised section 10.4.1 changed method blank criteria; 03/05/99.

20.2 Added Terms and Definitions section, Pollution Prevention, and Waste Management; 2/15/2000. Added Initial calibration %RSD criteria and linear regression criteria.

20.3 Changed name on cover from Monroe to Connecticut, no other changes required at this time; 8/16/00.

20.4 Added to Safety section.

20.5 Added to the Safety section and Waste Management sections. Added EH&S Officer to Approval section. Section 8.1 – added equivalent column. Section 11.4.1 Added soxtherm blank information. Section 9.6 – changed Aroclor-1242 to Aroclor-1016. Section 12.1 – added autosampler. Changed Tables 3.1, 4.0, and 4.1 to refer to LIM system for updated control limits. Section 18.1 revised wording of autosampler vial disposal. Section 12.3.2 added Soxtherm brief summary-1/19/2004.

20.6 May 7, 2004- Added 40% Rule for reporting of results to Section 12.7.

20.7 April 19, 2005- Section 9.6, Changed MS/MSD solution to be Aroclor 1016/1260 and updated Table 4.1.

20.8 April 19, 2005- Update Revision # and Date Effective.

20.9 April 19,2005-Added to Section 19, 8000C Reference.

20.10 May 15, 2007 – Section 5.2 – Added use of copper cleanup for sulfur. Sect. 8.1 – updated to use current columns. Sect 9.3 – changed from 6months to 1 year. Sect.10.2.1 – changed from .995 to 0.990 correlation coefficient. Sect 10.8.3 – Added return to control documenting. Sect 11.7.1 – files now scanned, not filed. Sect. 11.7.2 removed filing system section. Sect 11.8.5 – changed CAR procedure to electronic. Sect 11.8.7 – Removed reference to Labnet. Sect 11.9.2 – Changed from QCAR to Lims checklist for $1^{st}/2^{nd}$ level review. Sect 12.1 – updated to 6890's, columns, temperatures and ramps. Sect 12.6.1 – Removed that 2 analyses shall not be submitted. Sect 12.7.2 – Added to document a return to control. Sect 12.8.1 – Added Chemstation. Sect 12.8.3 – Changed manual integration signing. Sect 15.1 – changed reference from Labnet to Lims. Table 5.0 – added Aroclor-1262 and Aroclor-1268 as options.

20.11 Sept. 10, 2007- Sect 10.2.2 – added on choosing best fit for linear regression. Sect 5.3.2 and 5.3.3 removed on silica gel and florisil cleanup. Sect 6.2 – footnote 1 removed. Sect 7.1 – added caps. Sect 8.1 – added reference to sect 12.1. Sect 9.4.1 – added preparation to tables 1.0 and 2.0. Sect 12.3.1 – removed mecl2/acetone reference. Sect 10.3.2 added for 2^{nd} sources. Sect. 11.8.5 updated process.

Table 1.0 (preparation)

AR1660 Mixes

Aroclor 1016/1260 Intermediate	Initial Conc ug/ml	Amount Used mls	Final Volume mls	Final Conc. ug/ml	G_1660_INT_
Ar1016/1260 (G_1660_STK_)	1000	0.5	10	50	

TCX/DCB Intermediate	Initial Conc ug/ml	Amount Used mls	Final Volume mls	Final Conc. ug/ml	GSURINT_
TCX (G_TCX_STK_)	200	0.625	25	5	
DCB (G_DCB_STK_)	200	1.25	25	10	

Ar1660 Mix0.5	Initial Conc.	Amount Used	Final Volume	Final Conc.	Made as needed
G1660WRK1_	0.05	0.1	0.2	0.025	
Surrogate (concentration)				0.0025	DCB 2x higher

Ar1660 Mix1	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1660WRK1_
Ar1660Int (G_1660_INT_)	50	0.1	100	0.050	
SurrInt (GSURINT_)	5.0	0.1	100	0.005	DCB 2x higher

Ar1660 Mix2	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1660WRK2_
Ar1660Int (G_1660_INT_)	50	0.2	100	0.1000	
SurrInt (GSURINT_)	5.0	0.2	100	0.0100	DCB 2x higher

Ar1660 Mix3	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1660WRK3_
Ar1660Int (G_1660_INT_)	50	0.4	100	0.2000	
SurrInt (GSURINT_)	5.0	0.5	100	0.0250	DCB 2x higher

Ar1660 Mix4	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1660WRK4_
Ar1660Int (G_1660_INT_)	50	0.8	100	0.4000	
SurrInt (GSURINT_)	5.0	1	100	0.0500	DCB 2x higher

Ar1660 Mix5	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1660WRK5_
Ar1660Int (G_1660_INT_)	50	1.6	100	0.8000	
SurrInt (GSURINT_)	5.0	2	100	0.1000	DCB 2x higher

Ar1660- Mix3 ICV	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1660ICV_
Ar1016 (G_1016_SS_)	1000	0.01	50	0.20	
Ar1260 (G_1260_SS_)	1000	0.01	50	0.20	
SurrInt (GSURINT_)	5.0	0.25	50	0.025	DCB 2x higher

PCB Mixes

Aroclor 1221 Intermediate	Initial Conc ug/ml	Amount Used mls	Final Volume mls	Final Conc. ug/ml	G_1221_INT_
Ar1221 (G_1221_STK_)	1000	0.5	10	50	Restek 32007

Ar1221 Mix 2	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1221WRK2_
Ar1221 Int (G_1221_INT_)	50	0.2	50	0.20	
SurrInt (GSURINT_)	5	0.1	50	0.010	DCB 2x higher

Aroclor 1232 Intermediate	Initial Conc ug/ml	Amount Used mls	Final Volume mls	Final Conc. ug/ml	G_1232_INT_
Ar1232 (G_12321_STK_)	1000	0.5	10	50	Restek 32008

Ar1232 Mix 2	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1232WRK2_
Ar1232 Int (G_1232_INT_)	50	0.1	50	0.10	
SurrInt (GSURINT_)	5	0.1	50	0.010	DCB 2x higher

Aroclor 1242 Intermediate	Initial Conc ug/ml	Amount Used mls	Final Volume mls	Final Conc. ug/ml	G_1242_INT_
Ar1242 (G_1242_STK_)	1000	0.5	10	50	Restek 32009

Ar1242 Mix 2	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1242WRK2_
Ar1242 Int (G_1242_INT_)	50	0.1	50	0.10	
SurrInt (GSURINT_)	5	0.1	50	0.010	DCB 2x higher

Aroclor 1248 Intermediate	Initial Conc ug/ml	Amount Used mls	Final Volume mls	Final Conc. ug/ml	G_1248_INT_
Ar1248 (G_1248_STK_)	1000	0.5	10	50	Restek 32010

Ar1248 Mix 2	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1248WRK2_
Ar1248 Int (G_1248_INT_)	50	0.1	50	0.10	
SurrInt (GSURINT_)	5	0.1	50	0.010	DCB 2x higher

Aroclor 1254 Intermediate	Initial Conc	Amount	Final	Final Conc.	G_1254_INT_

	ug/ml	Used mls	Volume mls	ug/ml	
Ar1254 (G_1254_STK_)	1000	0.5	10	50	Restek 32011

Ar1254 Mix 2	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1254WRK2_
Ar1254 Int (G_1254_INT_)	50	0.1	50	0.10	
SurrInt (GSURINT_)	5	0.1	50	0.010	DCB 2x higher

Aroclor 1262 Intermediate	Initial Conc ug/ml	Amount Used mls	Final Volume mls	Final Conc. ug/ml	G_1262_INT_
Ar1262 (G_1262_STK_)	1000	0.5	10	50	Restek 32409

Ar1262 Mix 2	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1262WRK2_
Ar1262 Int (G_1262_INT_)	50	0.1	50	0.10	
SurrInt (GSURINT_)	5	0.1	50	0.010	DCB 2x higher

Aroclor 1268 Intermediate	Initial Conc ug/ml	Amount Used mls	Final Volume mls	Final Conc. ug/ml	G_1268_INT_
Ar1268 (G_1268_STK_)	1000	0.5	10	50	Restek 32410

Ar1268 Mix 2	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1268WRK2_
Ar1268 Int (G_1268_INT_)	50	0.1	50	0.10	
SurrInt (GSURINT_)	5	0.1	50	0.010	DCB 2x higher

TCX/DCB Intermediate	Initial Conc ug/ml	Amount Used mls	Final Volume mls	Final Conc. ug/ml	GSURINT_
TCX (G_TCX_STK_)	200	0.625	25	5	
DCB (G_DCB_STK_)	200	1.25	25	10	

TABLE 2.0
MULTI-COMPONENT
CALIBRATION CONCENTRATIONS, UG/ML

Analyte	Mix 0.5	Mix 1	Mix 2	Mix 3	Mix 4	Mix 5
Aroclor-1016/1260	0.025	0.05	0.10	0.20	0.40	0.80
Aroclor-1221			0.20			
Aroclor-1232			0.10			
Aroclor-1242			0.10			
Aroclor-1248			0.10			
Aroclor-1254			0.10			
Aroclor-1262			0.10			
Aroclor-1268			0.10			
TCX	0.0025	0.005	0.01	0.025	0.05	0.10
DCB	0.005	0.01	0.02	0.050	0.10	0.20

TABLE 3.0
SURROGATE MIX, UG/ML

Surrogate	Stock	Mix for Extractions (added to each sample and spike)
TCX	2.0	0.20
DCB	2.0	0.20

TABLE 3.1
SURROGATE RECOVERY LIMITS

Surrogate recovery limits are entered into the Laboratory Information Management system.

TABLE 4.0
AROCLOR QC CHECK SOLUTION (UG/ML)

Compound	Stock	STD Mix for Extractions (Added to each Aroclor QC Check Sample)	Water Control Limits	Soil Control Limits
Aroclor-1016	1000	5	See LIMS for updated limits	See LIMS for updated limits
Aroclor-1260	1000	5	See LIMS for updated limits	See LIMS for updated limits

TABLE 4.1
AROCLOR MATRIX SPIKE SOLUTION (UG/ML)

Compound	Stock	STD Mix for Extractions (Added to each Aroclor Matrix Spike Sample)	Water Control Limits	Soil Control Limits
Aroclor – 1016	1000	2.0	See LIMS for Updated limits	See LIMS for Updated limits
Aroclor-1260	1000	2.0	See LIMS for updated limits	See LIMS for updated limits

TABLE 5.0
PRACTICAL QUANTITATION LIMITS (PQL)

Analyte	Quantitation Limits, Water ug/L	Quantitation Limits, Soil ug/Kg
Aroclor-1016	0.50	17
Aroclor-1221	1.0	33
Aroclor-1232	0.50	17
Aroclor-1242	0.50	17
Aroclor-1248	0.50	17
Aroclor-1254	0.50	17
Aroclor-1260	0.50	17
Aroclor-1262 *	0.50	17
Aroclor-1268 *	0.50	17

- Additional compounds require special client request.

Figure 1.D

STL - Connecticut

GC SEMIVOLATILES INJECTION LOG Instr. hp5890-1d.i

Standards Codes:	Routine Maintenance Performed:	Date: 11-MAY-2007 10:55
	Cut & Cleaned: ( )	QC Batch: D1679.b
	Changed Sleeve: ( )	
	Other: ( )	

Stephan Z. Daren 05/14/07

Data File	Client ID	Sample ID	ALS	DF	Analyst	Reagent	Comments
D1679071.d	AR16603 0.2ng YSFS	AR16603 0.2ng	3	1	turbo1	42813	
D1679072.d	AR16603 0.2ng	AR16603 0.2ng	4	1	turbo1		
D1679073.d	PIBLK	PIBLK	5	1	turbo1	28337	
D1679074.d	VS-45-15R(10.0-10.5	220-1420-A-5-A	6	50	turbo1		RR @ 1:500
D1679075.d	P1-S119(6.0-6.5)	220-1420-A-26-A	7	50	turbo1		RR @ 1:10
D1679076.d	MB 220-5806/1-AA	MB 220-5806/1-AA	8	1	turbo1		
D1679077.d	LCS 220-5806/2-AA	LCS 220-5806/2-AA	9	1	turbo1		
D1679078.d	MB 220-5807/1-AA	MB 220-5807/1-AA	10	1	turbo1		
D1679079.d	LCS 220-5807/2-AA	LCS 220-5807/2-AA	11	1	turbo1		
D1679080.d	VS-45-15R(10.0-10.5	220-1420-A-5-A	12	500	turbo1		
D1679081.d	P1-S119(6.0-6.5)	220-1420-A-26-A	13	10	turbo1		RR STR
D1679082.d	P1-S119(6.0-6.5)	220-1420-A-26-A	14	1	turbo1		
D1679083.d	MB 220-5931/1-AA	MB 220-5931/1-AA	15	1	turbo1		
D1679084.d	LCS 220-5931/3-AA	LCS 220-5931/3-AA	16	1	turbo1		
D1679085.d	WC-210114/16-24	220-1468-A-1-G	17	1	turbo1		
D1679086.d	WC-210114/24-32	220-1468-B-2-E	18	1	turbo1		
D1679087.d	WC-210113/8-16	220-1468-A-3-K	19	1	turbo1		
D1679088.d	WC-210113/16-24	220-1468-B-4-D	20	1	turbo1		
D1679089.d	WC-210113/24-32	220-1468-C-5-C	21	1	turbo1		
D1679090.d	WC-210112/8-16	220-1468-C-6-D	22	1	turbo1		
D1679091.d	WC-210112/16-24	220-1468-C-7-C	23	1	turbo1		
D1679092.d	WC-210112/24-32	220-1468-A-8-C	24	1	turbo1		
D1679093.d	WC-210111/8-16	220-1468-B-9-B	25	1	turbo1		
D1679094.d	AR16603 0.2ng YSFS	AR16603 0.2ng	26	1	turbo1		
D1679095.d	PIBLK	PIBLK	27	1	turbo1		
D1679096.d	AR16603 0.2ng	AR16603 0.2ng	28	1	turbo1		

Witnessed By: [signature] Date: 5/14/07 Page No. _____

Figure 2.0

May, Dawn

Subject: 220-1461 Roux 05/15/07
Start Date: Tuesday, May 15, 2007
Due Date: Wednesday, May 16, 2007

Status: In Progress
Percent Complete: 0%

Total Work: 0 hours
Actual Work: 0 hours

Owner: Maturo, Kim

SEVERN TRENT STL

NON-CONFORMANCE/CORRECTIVE ACTION FORM

A. Originator Information **Client Inquiry:**

Client:	*Roux*	*Job Number:* *Sample number(s):*	*220-1461* *#4 TCLP*
Date/time Initiated:	*5/15/07*	*Fraction:*	*TCLP Pest*
Lab/Client Originator:	*Kim Maturo*	*Date/time response due:*	*5/16/07*

Groups Involved:

	Sample control		Wet Chemistry		Metals	X	Organic Extractions
X	Gas Chromatography		MS-VOA		MS-SV		Report Generation
X	Client Services		Subcontractor				EDD

Detailed Description of Potential Problem:
Both surrogates failed VERY low in the sample and it therefore requires reextraction as soon as possible since the job is late. Sarah-Please let Dan know ASAP if there isn't sufficient leachate. 5/15/07 KM

B. Quality Assurance Information
Recommended Corrective Action:

C. Final Resolution
Describe What Happened and Corrective Action Taken:

Supervisor Review: Date: Date/time of Client notification:

Date EDD/Report Revision Sent:_____

D. Quality Assurance Final Approval (QA Manager or designee use only)

Corrective Action Approved:_____

STL Doc.# QAF00204.CT

Figure 3.0

STL Connecticut

LIMS Organics Data Review Checklist

Batched/First Level	Second Level
Prep Batch created (VOA) -TCLP's/MeOH Preps	
Check Method	
Dilutions appropriate	
Q-Editing checked	
TIC's called	Recheck
AD Worksheet (VOA) Init/FV	Recheck
AD Reagents (VOA) codes/amts	Recheck
Sample Results -RL's look correct -Flagging correct -RE/DL suffixes as needed	Recheck
GC Dual Column criteria applied -P/A sample results -P/S surrogates	Recheck
Surrogate recoveries present/flagged	Recheck
TIC's reported correctly	Recheck
QC linking correct	Recheck
NCM's created (record batch#)	Read

JOB LOCKED (PM Desktop)

Doc# QAF04300.CT

GAS CHROMATOGRAPHY INSTRUMENT MAINTENANCE LOG

Instrument ID ____

Date	*Analyst*	*Problem*	*Maintenance Performed*

Instrument ID ____

Date	*Analyst*	*Problem*	*Maintenance Performed*

Instrument ID ____

Date	*Analyst*	*Problem*	*Maintenance Performed*

Instrument ID ____

Date	*Analyst*	*Problem*	*Maintenance Performed*

FIGURE 4.0

Title: SOP for Metals Digestion -Soils
[Method SW846 3050B]

Approvals (Signature/Date):

Technical Manager	Date	Health & Safety Manager / Coordinator	Date
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. MES01009.CT.

Any reference within this document to Severn Trent Laboratories, Inc. or STL, should be understood to refer to TestAmerica Laboratories, Inc. (formerly known as Severn Trent Laboratories, Inc.)

Facility Distribution No. _____ Distributed To:_____

1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 This document is used to specifically describe the digestion of soils, sediments and sludge samples for analysis by ICP-MS and ICP-OES. Samples prepared by these methods may be analyzed for the listed metals. Users should be aware that this digestion is not a total digestion for most samples. If absolute total digestion is required, then alternate methods should be utilized.

2.2 Elements

Aluminum	Magnesium	Arsenic
Antimony	Manganese	Selenium
Barium	Molybdenum	Thallium
Beryllium	Nickel	Boron
Cadmium	Potassium	Strontium
Calcium	Silver	Zirconium
Chromium	Sodium	Mercury (ICP-MS)
Cobalt	Tin	
Copper	Titanium	
Iron	Vanadium	
Lead	Zinc	

2.3 The document control number for this SOP is CT-MES-10, rev 10.

3.0 TERMS AND DEFINITIONS

3.1 There are many definitions used with in the laboratory, which may be generic to all laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used with in the laboratory, reference the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 A representative 1.5 - 2 gm (wet weight) sample is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with hydrochloric acid and brought to 100 ml final volume with deionized water.

4.2 This method is based on EPA method 3050B.

5.0 INTERFERENCES

5.1 Samples of varying matrices provide different interferences. A list of quality controls are performed to evaluate these and are described in section 11.0.

5.2 Contamination can be a problem during sample preparation and must be minimized. Clean all work surfaces daily with DI water including bench-tops, hood and hot blocks. Take care not to cross contaminate samples during digestion by making sure that none of the samples boil or splatter.

5.3 Gold chloride is added to the digestates for analysis by ICP-MS. This is to stabilize the mercury and prevent its deposition and subsequent release from the sample introduction system. Gold chloride also increases the solubility of silver in nitric acid thus giving the added benefit of stabilizing silver.

6.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

6.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

6.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposur Limit (2	Signs and symptoms of exposure

Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contac may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TW 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and sever skin burns. Concentrated solutions cause deep ulcer and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permaner eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

7.1 Samples are collected in glass soil jars and refrigerated upon receipt.

7.2 Samples are stable when digestion is complete and need no preservation.

7.3 Holding time is 180 days.

8.0 APPARATUS AND MATERIALS

8.1 100 mL graduated digestion tubes – Environmental Express SC490 (single use only).

8.1.1 Matching ribbed watch glasses (single use only).

8.2 Environmental Express Hot Block Digestion Unit (SC100)

8.3 Calibrated Thermometer

8.4 Eppendorf pipets (Brinkmann 2000 series, variable, 100uL and 1000uL).

8.5 Mortar and pestle

8.6 Balance calibration weights

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Barnstead deionized water, 16.6 megohm or higher

9.2 Concentrated Nitric Acid, Trace Grade

9.3 Concentrated Hydrochloric Acid, Trace Grade

9.4 30% Hydrogen Peroxide

9.5 The sample spike receives an aliquot of matrix spike solution purchased from Inorganic Ventures.

CLPP-SPK-1

Analyte	Stock Conc. mg/L	Stock Vol. mL	Sample Vol. mL	Final Conc. ug/L
Al, Ba	2000	0.05	100	1000
Fe	1000	0.05	100	500
Co, Mn, Ni, V, Z	500	0.05	100	250
Cu	250	0.05	100	125
Cr	200	0.05	100	100
Ag, Be	50	0.05	100	25

CLPP-SPK-5

Analyte	Stock Conc. mg/L	Stock Vol. mL	SampleVol. mL	Final Conc. ug/L
Sb	100	0.10	100	100
Cd, Se, Tl	50	0.10	100	50
As	40	0.10	100	40
Pb	20	0.10	100	20

Earth Metals Spk

Analyte	Stock Conc. mg/L	Stock Vol. mL	Sample Vol. mL	Final Conc. ug/L
Ca, K, Mg, Na	5000	0.10	100	5000

9.6 Laboratory Control Sample Stock purchased from Inorganic Venture. Expiration date is one year from date of manufacture.

2007ICS-1

Analyte	Stock Conc. mg/L	Stock Vol. mL	Final Vol. mL	Final Conc. ug/L
Ti	1000	0.250	100	2500
B	500	0.250	100	1250
Mo	300	0.250	100	750
Si	230	0.250	100	575

2007ICS-2

Analyte	Stock Conc. mg/L	Stock Vol. mL	Final Vol. mL	Final Conc. ug/L
Sb	1000	0.25	100	2500

200ICS-3

Analyte	Stock Conc. mg/L	Stock Vol. mL	Final Vol. mL	Final Conc. ug/L
K	20000	0.250	100	50000
As, Pb, Tl	1000	0.250	100	2500
Se	500	0.250	100	1250
Ag, Ba, Cd, Co, C Cu, Ni, V, Zn	300	0.250	100	750
Mn	200	0.250	100	500
Be	100	0.250	100	250

200ICS-4

Analyte	Stock Conc. mg/L	Stock Vol. mL	Final Vol. mL	Final Conc. ug/L
Ca	15000	0.500	100	75000
Fe	12500	0.500	100	62500
Na	2500	0.500	100	12500
Mg	7500	0.500	100	37500
Al	3000	0.500	100	15000

Na Stock

Analyte	Stock Conc. mg/L	Stock Vol. mL	Final Vol. mL	Final Conc. ug/L
Na	10000	0.625	100	62500

9.7 1000 ppm AuCl3 solution. Dilute 1 gram of AuCl3 (99.9+%) to 1 L with deionized water.

10.0 CALIBRATION

10.1 The balance shall be calibrated each day used. This is done by first zeroing the balance and then placing a known standard weight of 1.0 grams on the balance and allowing the balance to stabilize. Enter the amount of the weight and press enter. The balance is now calibrated.

10.2 Eppendorf Pipets (Brinkmann 2000 series, variable, 100uL, 1000uL.

Eppendorf pipets must be calibrated quarterly at similar volumes used to dispense standards and reagents.

Reagent water stored at room temperature is pipetted into a tared disposable beaker. The milliliters dispensed should equal the weight in grams. Pipets used for standards should be accurate to 0.5% and those used for reagents should be accurate to 1%. If not, calibrate the eppendorf following the manufacturer's instructions. Calibrations are to be recorded in a log specific to each pipet.

10.3 Hot Block Digestion Unit (SC100)

The temperature of the digestor must be monitored during each batch. This is done by filling a digestion tube ¾ full with reagent water and placing it in a digestion slot. A calibrated thermometer is then placed in the tube and monitored throughout the digestion. The temperature

temperature is recorded at the beginning and end of the cycle in LIMS.

11.0 QUALITY CONTROL

11.1 Preparation blanks are carried throughout the entire sample and analytical process to check for contamination

11.2 A lab control sample is prepared one per 20 samples to check that no analyte is lost or gained during the batch preparation.

11.3 A duplicate sample is prepared one per 20 samples and the RPD is calculated from the original sample.

11.4 A sample spike is prepared one per 20 samples and the %R is calculated from the original sample.

11.5 QC related Activities

Prep. Blank	Every Batch not to exceed 20 samples
Lab Control Sample	Every Batch not to exceed 20 samples
Sample Spike	Every Batch not to exceed 20 samples
Duplicate	Every Batch not to exceed 20 samples

12.0 SAMPLE PREPARATION

12.10 Mix the sample thoroughly to achieve homogeneity, reference STL SOP for homogenization and compositing. Place 1.5 - 2 grams of wet sample weighed to the nearest 0.01g in the bottom of a 100 ml disposable digestion tube, making sure that little or no sample is stuck to the sides of the tube. Add 5 ml of DI water followed by 5 ml of concentrated nitric acid. Swirl to mix the slurry, and cover with a disposable tube cover. Heat the sample in a block digester at 95° C ± 5 °C and reflux for 10 minutes without boiling.

12.11 Allow the sample to cool, add 5 ml of concentrated nitric acid, replace the cover and reflux for an additional 30 minutes. Do not allow the volume to be reduced to less than 5 ml.

12.12 Cool the sample and add 2 ml of DI water and 3 ml of 30% hydrogen peroxide. Cover and return the tube to the heat source for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessive vigorous effervescence. Heat until effervescence subsides, and cool the tube.

12.13 Continue to add peroxide in 1 ml aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. Do not add more than a total of 6 ml of

peroxide. When adding additional amounts of peroxide, always add the same amount to the preparation blank and laboratory control sample. Cover and return the hydrogen peroxide digestate to the heat source for two hours or until the digestate reaches approximately 5 mL, making sure that the sample does not boil or splatter.

12.14 Cool the sample, add 5 ml of concentrated HCl, cover and return the tube to the digester. Heat for an additional 15 minutes. After the sample has cooled add DI water to the 100 ml mark. Add 0.1 ml 1000 ppm AuCl3 solution to all samples including QC, cap tightly and shake well. The sample is now ready for analysis.

13.0 CALCULATIONS

13.1 None.

14.0 ACCEPTANCE OF DATA

14.1 N/A

15.0 REPORTING OF RESULTS

15.1 Samples are entered into LIMS with all prep information.

15.2 A batch system is used. Only 20 samples are allowed in a batch. Each batch should have a duplicate sample, a spiked sample, a blank sample and a laboratory control sample. All of these samples should be indicated in LIMS.

15.3 Record weight (to nearest 0.01 gram), matrix, initial volume, color and clarity and final volume for each individual sample.

15.4 Record lot number of all solutions and reagents added.

15.5 Record in comment column if sample is diluted, concentrated, limited, or other changes.

16.0 POLLUTION PREVENTION

16.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

16.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable

amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

16.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

16.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

16.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

16.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

17.0 WASTE MANAGEMENT

17.1 All waste shall be managed in accordance with all state and federal requirements. See the Testamerica-CT Hazardous Waste Management Plan.

17.2 All personnel who handle or generate waste must be trained within six months of employment in proper waste handling and requirements.

18.0 SUPPLEMENTAL DOCUMENTS

18.1 **None**

19.0 REFERENCES

19.1 Method 3050B, SW-846, 3rd ed., Test Methods for Evaluating Solid Waste, EPA Office of Solid Waste and Emergency Response, Dec. 1996.

19.3 Employee Chemical Safety Handbook

20.0 SUBSTANTIVE REVISIONS

20.1 Original issue - 4/21/93

20.2 Modified sections 3.1 and 11.5 to include As, Cd, Pb, Se, and Tl to ICAP analysis for trace ICP.

20.3 Changed name to STL from IEA.

20.4 Added section 3 – Terms and definitions, 16 & 17 for waste management and pollution prevention, renumber other sections; 02/12/00.

20.5 Modified sections 8.1 and section 12 to include the use of hot block March 20, 2001.

20.6 Updated for labnet in section 15; added eppendorf and hotblock calibration in section 10; March 15, 2003.

20.7 Updated section 6 to include the corporate health and safety SOP January 28, 2004.

20.8 Added spiking concentration table to section 9.7, April 1, 2004.

20.9 Updated section 12.10: to include reference to SOP on sample homogenization; correct sample weight.

20.10 Update section 12.11 and 12.21 to include nitric acid step.

20.11 Update section 12.13 and 12.23 to include hydrogen peroxide step.

20.12 Modify section 12.14 to change HCL digestion time from 10 minutes to 15 minutes.

20.13 Modify section 12.24 to change final volume from 200 mL to 100 mL.

20.14 Modify section 12.24 to change final volume from 100 mL to 50 mL.

20.15 Removed section 12.2. Hotblock is no longer used.

20.16 Added the use of AuCl3 as a stabilizer for Hg analysis by ICP-MS.

20.17 Added new TestAmerica SOP header and control number, changed name, 01/16/08.

Title: SOP for Metals Digestion- Aqueous
[Method SW846 3010A]

Approvals (Signature/Date):

Technical Manager	Date	Health & Safety Manager / Coordinator	Date
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. MES00909.CT.

Any reference within this document to Severn Trent Laboratories, Inc. or STL, should be understood to refer to TestAmerica Laboratories, Inc. (formerly known as Severn Trent Laboratories, Inc.)

Facility Distribution No. _____ Distributed To:_____

1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 This digestion procedure is used for the preparation of aqueous samples for analysis by ICP, for the metals listed below. The procedure is used to determine total metals for unfiltered samples that are HNO3 preserved to a pH less than 2.

2.2

Aluminum	Lead	Titanium
Antimony	Magnesium	Vanadium
Arsenic	Manganese	Zinc
Barium	Molybdenum	Boron
Beryllium	Nickel	Strontium
Cadmium	Potassium	Silicon
Calcium	Selenium	Zirconium
Chromium	Silver	
Cobalt	Sodium	
Copper	Thallium	
Iron	Tin	

2.3 The document control number for the SOP is CT-MES-9, rev 10.

3.0 TERMS AND DEFINITIONS

3.1 There are many definitions used with in the laboratory, which may be generic to all laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used with in the laboratory, reference the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 A mixture of HNO_3 and material to be analyzed is heated in a 50mL or 100 mL tube until the volume is reduced to 5 mL. After cooling, the digestion is refluxed with HCl and brought back to the original volume. If the sample should go to dryness at any time during the digestion, it must be discarded and the sample reprepared.

4.2 This method is based on EPA method 3010A.

4.3 This method deviates from method 3010A to include antimony and silver. A laboratory control sample containing antimony and silver is digested and analyzed to check that no analyte is lost or gained during the preparation.

5.0 INTERFERENCES

5.1 Samples of varying matrices provide different interferences. A list of quality controls are performed to evaluate these and are described in section 10.0.

5.2 Contamination can be a problem during sample preparation and must be minimized. Clean all work surfaces daily with DI water including bench tops, hood and hot plates. Take care not to cross contaminate samples during digestion by making sure that none of the samples boil or splatter.

6.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

6.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

6.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of `materials` used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (	Hazards	Exposure Limit (2)	Signs and symptoms of exposure

Hydrochlor Acid	Corrosive Poison	5 ppm-Ceili	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contac may cause severe burns and permanent eye damage.
Nitric Aci	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and sever skin burns. Concentrated solutions cause deep ulcer and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permaner eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

7.1 Samples are collected in 500 mL plastic bottles acidified to pH of < 2 with nitric acid.

7.2 Samples are stable when digestion is complete and need no preservation. Sample bottles are not to be reused.

7.3 Holding time is 180 days.

8.0 APPARATUS AND MATERIALS

8.1 Equipment

8.1.1 Ventilation Hood
8.1.2 Environmental Express Hot Block Digestor, model SC100
8.1.3 Environmental Express certified digestion tubes
8.1.4 Watch glasses
8.1.5 Eppenfdorf pipette, 1000uL, 100uL

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Barnstead deionized water, 16.6 megohm or higher

9.2 Concentrated Nitric Acid, Trace Grade

9.3 Concentrated Hydrochloric acid, Trace Grade

9.4 Sample matrix spike stock purchased from Inorganic Ventures. For matrix spike analysis, add appropriate amount of each spike stock directly to the designated QC sample. Expiration date is one year from date of manufacture.

CLPP-SPK-1

Analyte	Stock Conc. mg/L	Stock Vol. mL	Sample Vol. mL	Final Conc. ug/L
Al, Ba	2000	0.10	100	2000
Fe	1000	0.10	100	1000
Co, Mn, Ni, V, Z	500	0.10	100	500
Cu	250	0.10	100	250
Cr	200	0.10	100	200
Ag, Be	50	0.10	100	50

CLPP-SPK-5

Analyte	Stock Conc. mg/L	Stock Vol. mL	SampleVol. mL	Final Conc. ug/L
Sb	100	0.10	100	100
Cd, Se, Tl	50	0.10	100	50
As	40	0.10	100	40
Pb	20	0.10	100	20

Earth Metals Spk

Analyte	Stock Conc. mg/L	Stock Vol. mL	Sample Vol. mL	Final Conc. ug/L
Ca, K, Mg, Na	5000	0.20	100	10000

9.5 Laboratory Control Sample Stock purchased from Inorganic Venture. Expiration date is one year from date of manufacture.

2007ICS-1

Analyte	Stock Conc. mg/L	Stock Vol. mL	Final Vol. mL	Final Conc. ug/L
Ti	1000	1.00	1000	1000
B	500	1.00	1000	500
Mo	300	1.00	1000	300
Si	230	1.00	1000	230

2007ICS-2

Analyte	Stock Conc. mg/L	Stock Vol. mL	Final Vol. mL	Final Conc. ug/L
Sb	1000	1.00	1000	1000

200ICS-3

Analyte	Stock Conc. mg/L	Stock Vol. mL	Final Vol. mL	Final Conc. ug/L
K	20000	1.00	1000	20000
As, Pb, Tl	1000	1.00	1000	1000
Se	500	1.00	1000	500
Ag, Ba, Cd, Co, C Cu, Ni, V, Zn	300	1.00	1000	300
Mn	200	1.00	1000	200
Be	100	1.00	1000	100

200ICS-4

Analyte	Stock Conc. mg/L	Stock Vol. mL	Final Vol. mL	Final Conc. ug/L
Ca	15000	2.00	1000	30000
Fe	12500	2.00	1000	25000
Na	2500	2.00	1000	5000
Mg	7500	2.00	1000	15000
Al	3000	2.00	1000	6000

Na Stock

Analyte	Stock Conc. mg/L	Stock Vol. mL	Final Vol. mL	Final Conc. ug/L
Na	10000	2.50	1000	25000

10.0 CALIBRATION

10.1 Eppendorf Pipets

Eppendorf pipets must be calibrated quarterly at similar volumes used to dispense standards and reagents.

Reagent water stored at room temperature is pipetted into a tared disposable beaker. The milliliters dispensed should equal the weight in grams. Pipets used for standards should be accurate to 0.5% and those used for reagents should be accurate to 1%. If not, calibrate the eppendorf following the manufacturer's instructions. Calibrations are to be recorded in a log specific to each pipet.

10.2 Hot Block Digestion Unit (SC100)

The temperature of the digestor must be monitored during each batch. This is done by filling a digestion tube ¾ full with reagent water and placing it in a digestion slot. A calibrated thermometer is then placed in the tube and monitored throughout the digestion. The temperature is recorded at the beginning and end of the cycle in labnet.

11.0 QUALITY CONTROL

11.1 Preparation blanks are processed one per 20 samples through all steps of the digestion and are performed to check for contamination

11.2 A laboratory control sample is prepared one per 20 samples to check that no analyte is lost or

gained during the batch preparation.

11.3 A duplicate sample is prepared one per 20 samples and the RPD is calculated from the original sample.

11.4 A sample spike is also performed one per 20 samples with each batch.

11.5 QC related Activities

Preparation Blank	Every Batch not to exceed 20 samples
Laboratory Control Sample	Every Batch not to exceed 20 samples
Sample Spike	Every Batch not to exceed 20 samples
Duplicate	Every Batch not to exceed 20 samples

12.0 SAMPLE PREPARATION

12.1 Hot Block Digestion

Shake the sample well and immediately pour it into a graduated 50 mL or 100 mL hot block tube. For QC samples, additional aliquots are needed, shake the sample between each subsequent addition. Add stock spike to the QC sample as described in section 9.4. For LCS analysis, treat the LCS solution like a sample using an aliquot of the solution prepared in section 9.5. Add 3% of concentrated HNO_3 to the tube and place it in a hotblock. Heat the sample until the volume is reduced to 5 ml, making certain that the sample does not boil and that no portion of the bottom of the tube is allowed to go dry. Discard and reprepare any sample that goes to dryness.

Cool the tube and add 1% of concentrated HCl. Allow the sample to reflux for 15 minutes. Remove the tube from the heat source and while still warm, rinse down the sides of the tube with reagent water. Adjust to final volume with reagent water, close cap and mix by inverting several times. The sample is now ready for analysis.

13.0 CALCULATIONS

13.1 None.

14.0 ACCEPTANCE OF DATA

14.1 Acceptance of the data is described in section 10.0

15.0 REPORTING OF RESULTS

15.1 Samples are entered into the LIMS system. All appropriate sections must be completed. The data prep page should be printed and put in job folder.

15.2 A batch system is used. Only 20 samples are allowed in a batch. Each batch should have a duplicate sample, a spiked sample, spiked duplicate, a prep-blank and a laboratory control sample. All of these samples should be indicated in the system logbook page.

15.3 Record matrix, initial volume and final volume.

15.4 Record lot number of all solutions and reagents added.

15.5 Record in comment column if sample is diluted, concentrated, limited, preserved, reprepped or other changes.

16.0 POLLUTION PREVENTION

16.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

16.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

16.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

16.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

16.1.4 Waste Reduction; Reduce the volume of waste generated wherever possible.

16.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

17.0 WASTE MANAGEMENT

17.1 All waste shall be managed in accordance with all state and federal requirements. See the TESTAMERICA-CT Hazardous Waste Management Plan.

17.2 All personnel who handle or generate waste must be trained within six months of employment

employment in proper waste handling and requirements.

18.0 SUPPLEMENTAL DOCUMENTS

18.1 **None**

19.0 REFERENCES

19.1 Method 3010A, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy, July 1992.

19.2 Employee Chemical Safety Handbook

20.0 SUBSTANTIVE REVISIONS

20.1 Changed name to STL from IEA. Updated method reference

20.2 Added section 3 – Terms and definitions, 16 & 17 for waste management and pollution prevention, renumber other sections: 02/12/00.

20.3 Added the Hot block digestion procedure in section 12; added reference to labnet in section 15; added calibration of eppendorf and Hotblock digestor in section10.0: 03/310/3

20.4 Updated section 6 to include the corporate health and safety SOP: 01/28/2004.

20.5 Added spike and LCS tables to section 9.0,: 04/01/2004

20.6 Section 2.1: removed digestion of dissolved metals; 03/18/2005

20.7 Modified Section 12.1, HNO3 final volume 5%, HCL 1%; 09/19/2007.

20.8 Section 4.3: added addressed antimony and silver method deviation; 03/18/2005.

20.9 Added new TestAmerica SOP header and control number, changed name, 01/16/08.

Title: SOP for ICP Metals Analysis
[Method SW846 6010B]

Approvals (Signature/Date):

Technical Manager	Date 10/18/07	Health & Safety Manager / Coordinator	Date 10-22-07
Quality Assurance Manager	Date 10/22/07	Laboratory Director	Date 10/19/07

This SOP was previously identified as SOP No. MES02006.CT.

Facility Distribution No. _____ Distributed To: _____

Any reference within this document to Severn Trent Laboratories, Inc. or STL, should be understood to refer to TestAmerica Laboratories, Inc. (formerly known as Severn Trent Laboratories, Inc.)

1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 Inductively coupled plasma-atomic emission spectrometry determines trace elements, including metals, in solution. The method is applicable to aqueous samples, TCLP and SPLP extracts, industrial wastes, soils, sludges and sediments. All samples are digested following appropriate digestion methods.

2.2 Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences common to optical emission techniques.

2.3 The analytes determined using this technique are:

Aluminum	Cobalt	Potassium	Zinc
Antimony	Copper	Selenium	Boron
Arsenic	Iron	Silver	Silicon
Barium	Lead	Sodium	Strontium
Beryllium	Magnesium	Tin	Zirconium
Cadmium	Manganese	Titanium	
Calcium	Molybdenum	Thallium	
Chromium	Nickel	Vanadium	

2.4 The document control number for this SOP is CT-MES-20, Rev. 6.

3.0 TERMS AND DEFINITIONS

3.1 There are many definitions used with in the laboratory, which may be generic to all laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used with in the laboratory, reference the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 This method is used to analyze samples that have been previously digested for ICP analysis.

Samples are introduced into a cross flow nebulizer and spray chamber using a peristaltic pump. Element specific emission spectra are produced by a radio frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are measured. These intensities are compared to a standard curve and the concentration of each element is determined.

4.2 This method is based on SW846 method 6010B.

4.3 This method does not deviate from the SW846 method 6010B.

5.0 INTERFERENCES

Spectral interferences include overlaps from other elements, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena and background from stray light or high concentration elements. This is minimized by using interelement correction factors. Multiplication factors are set up to compensate for positive and negative results for all of the other elements. These factors will vary slightly over time and will change when major maintenance is done on the instrument. To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. A computer routine must be used for automatic correction on all determinations. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. These are set up by analyzing all of the elements in 2.3 individually at concentrations near the upper linear range. Background emission and stray light can be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. To determine the appropriate location for off-line background correction scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes.

When interelement corrections are applied, their accuracy should be verified, daily, by analyzing spectral interference check solutions. All interelement spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation change, such as in the torch, nebulizer, injector, or plasma conditions occurs. Standard solution should be inspected to ensure that there is no contamination that may be perceived as a spectral interference.

Physical interferences are associated with sample viscosity or high dissolved solids. Viscosity is minimized by using a peristaltic pump, and high solids are overcome by sample

dilution.

Chemical interferences are not normally pronounced in ICP and can be minimized by selection of proper operating conditions.

6.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

6.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

6.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

7.1 Samples are collected in 500 mL plastic bottles acidified to pH of < 2 with nitric acid.

7.2 Samples are stable when digestion is complete and need no preservation. Sample bottles are not to be reused.

7.3 Holding time is 180 days.

8.0 APPARATUS AND MATERIALS

8.1 Thermo Jarrell Ash (TJA) 61E Trace ICAP

8.2 Mass Flow Controller

8.3 Peristaltic Pump

8.4 High purity argon, welders grade or better

8.5 Thermo Jarrell Ash Autosampler Model AS 300

8.6 Personal Computer

8.7 100 and 1,000 uL variable Eppendorf pipettes

8.8 10 ml graduated serological pipettes

8.9 12 mL capacity polypropylene sample tubes (Borosilicate glass must not be used for the analysis of Boron or Silicon in order to avoid sample contamination.

8.10 Thermo Jarrel Ash Operator's Manual - February 1991 Part #128832.01

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Reagent water - ASTM Type II, 18 megohm, deionized

9.2 Trace grade nitric acid.

9.3 ICP stock standards: All standards are to be plasma grade and must come with certificates of analysis traceable to NIST standards. All analytes in the calibration verification standards must be from a different source than those found in the calibration standards. The following analyte combinations are to be used:

9.3.1 Calibration Standard 1: 100 ppm Ag, 1000 ppm each Al, As, Ba, Be, Cd, Cr, Co, Cu, Fe, Pb, Mn, Ni, Se, Sr, Tl, V and Zn.
9.3.2 Calibration Standard 2: 1000 ppm each Sb, B, Mo, Si, Sn, Ti and Zr.
9.3.3 Earth Metals Calibration Standard: 5000 ppm each Ca, Mg, K and Na.
9.3.4 1000 ppm Al Calibration Standard

9.3.5 1000 ppm Fe Calibration Standard
9.3.6 Calibration Verification Standard 1: 1000 ppm each Ca, Mg, K and Na, 500 ppm each Al, As, Ba, Be, Cd, Cr, Co, Cu, Fe, Pb, Mn, Ni, Se, Sr, Tl, V and Zn.
9.3.7 1000 ppm Al second source
9.3.8 1000 ppm Fe second source
9.3.9 10000 ppm Na
9.3.10 10000 ppm Ca
9.3.11 10000 ppm Mg
9.3.12 10000 ppm K
9.3.13 100 ppm Ag
9.3.14 Calibration Verification Standard 2: 500 ppm each Sb, B, Mo, Si and Ti.
9.3.15 Calibration Verification Standard 3: 500 ppm each Sn and Zr
9.3.16 CRI Standard: 500 ppm each Ca, Mg, K and Na, 20 ppm each Al and Ba, 10 ppm Fe, 6 ppm each Sb and Zn, 5 ppm each Co and V, 4 ppm Ni, 3.5 ppm Se, 2.5 ppm each Cu and Tl, 1.5 ppm each As and Mn, 1 ppm each Cr, Pb and Ag, 0.5 ppm each Be and Cd.
9.3.17 ICSA Standard: 5000 ppm each Ca, Mg, Al, and 2000 ppm Fe.
9.3.18 ICSAB Standard: 100 ppm each Cd, Ni and Zn, 60 ppm Sb, 50 ppm each Ba, Be, Cr, Co, Cu, Mn and V, 20 ppm Ag, 10 ppm each As and Tl and 5 ppm each Pb and Se.

9.4 Working Standards: All working standards are made in volumetric flasks with a final acid concentration of 6 % nitric. Standards that contain Boron or Silicon must be stored in plastic bottles.

9.4.1 Standard 1: Reagent water contained in a 1L plastic bottle. This solution is also used for sample dilution and all ICB/CCB's.
9.4.2 Standard 2: 100 ul of the standard from section 9.3.1 + 100 ul of the standard from section 9.3.2 + 1000 ul of the standard from section 9.3.3 + 1000 ul of the standard from section 9.3.4 + 1000 ul of the standard from section 9.3.5 to 100.0 ml.
9.4.3 ICV: 200 ul of the standard from section 9.3.6 + 200 ul of the standard from section 9.3.14 + 200 ul of the standard from section 9.3.15 + 1000 ul of the standard from section 9.3.7 + 1000 ul of the standard from section 9.3.8 + 250 ul each of the standards from sections 9.3.9, 9.3.10, 9.3.11 and 9.3.12 + 100 ul of the standard from section 9.3.13 to 100.0 ml.
9.4.4 CRI: 1000 ul of the standard from section 9.3.16 to 100.0 ml.
9.4.5 ICSA: 10.0 ml of the standard from section 9.3.17 to 100.0 ml.
9.4.6 ICSAB: 10.0 ml of the standard from section 9.3.17 + 1000 ul of the standard from section 9.3.18 to 100.0 ml.
9.4.7 CCV: 200 ul of the standard from section 9.3.6 +200 ul of the standard from section 9.3.14 + 200 ul of the standard from section 9.3.15 + 1000 ul of the standard from section 9.3.7 + 1000 ul of the standard from section 9.3.8 + 400 ul of the standard from section 9.3.9 + 375 ul of the standard from section 9.3.10 + 375 ul of the standard from section 9.3.11 + 200 ul of the standard from section 9.3.12 + 100 ul of the standard from section 9.3.13 to 200.0 ml.

10.0 CALIBRATION

10.1.1 Calibration must be performed once every 24 hours. The calibration sequence is as followed: calibration of standard 1, then calibration of standard 2. Quality control solutions are then analyzed to verify calibration. See section 11 for quality control solutions.

10.2 The average of two burns is used for all calibration, quality control, and client samples.

11.0 QUALITY CONTROL

11.1 The Initial Calibration Verification solution (ICV) is analyzed immediately following calibration. The results of the ICV are to agree within 10% of the true values. If not, terminate the analysis, correct the problem, and recalibrate the instrument.

11.2 Initial Calibration Blank (ICB) is analyzed immediately following the ICV. The results of the ICB are to be less than three times the MDL. If not, terminate the analysis, correct the problem, and recalibrate the instrument.

11.3 Low Level QC Check Standard (CRI) is analyzed at the beginning of each analytical run. The results of the CRI are to agree within 20% of the true values.

11.4 Interference Check Solutions (ICSA and ICSAB) are analyzed at the beginning of each analytical run to verify the interelement and background correction factors. The non-spiked B elements in the ICSA shall be less than two times the MDL, unless they are verified trace impurities. The spike results are to agree within 20% of the true values.

11.5 Continuing Calibration Verification (CCV) is analyzed prior to any samples, after every ten samples, and at the end of an analytical run. The results of the CCV are to agree within 10% of the true values. If not, terminate the analysis, correct the problem, recalibrate the instrument, and reanalyze all samples that are not bracketed by acceptable CCV's.

11.6 Continuing Calibration Blank (CCB) is analyzed immediately following the CCV, after every ten samples, and at the end of an analytical run. The results of the CCB are to be less than three times the MDL. If not, terminate the analysis, correct the problem, and recalibrate the instrument. Reanalyze all samples not bracketed by acceptable CCB's except those where analyte concentrations are greater than 10 times the CCB level for the affected analytes.

11.7 Method Blank (PB) is analyzed along with the sample batch it was prepared with. The PB must be carried through the entire procedure and contain the same acid concentration as the samples being analyzed. The results of the PB are to be less than one-half of reporting limit.

If not, redigest all samples in the associated batch and reanalyze the samples. This does not apply to samples where analyte concentrations are greater than 10 times the PB level for the affected analytes.

11.8 Laboratory Control Sample (LCS) is also carried through the entire procedure as the samples being analyzed. The results of the LCS are to agree within 20% of the true values for aqueous samples and within vendor specified limits for solids. The acceptance criteria for silver in soil is 75-120% or within vendor specified limits. If not, redigest all samples in the associated batch and reanalyze samples.

11.9 Matrix Duplicate is analyzed at a frequency of one per matrix batch that does not exceed 20 actual samples. A duplicate sample is brought through the entire sample preparation and analytical process in duplicate. A control limit of 20% RPD is used for sample values greater than ten times the MDL.

11.10 Matrix Spike is analyzed at a frequency of one per matrix batch that does not exceed 20 actual samples. The matrix spike concentrations can be referenced in STL CT SOP MES00907 section 9.4. For DOD QSM, The concentration of the spiked compounds shall be at or below the midpoint of the calibration range or at the appropriate level of concern. If the native concentration is known, the MS should be spiked 1–4 times that concentration, if the the amount of matrix spike added to the parent sample is not 1-4 times that of the parent sample concentration, then the matrix spike sample will be redigested and reanalyzed. . If repreparation and analysis are not feasible because of not enough samples or expired holding time, the failed MS data may be reported and flagged as not useable because of inappropriate spiking concentration.

11.11 Post Digestion Spike (PDS) is analyzed for every matrix spike prepared to check for matrix interferences. A portion of digested sample is spiked with the same spiking solutions as are used for digestion. A control limit of 25% of the true values is used for sample values less than four times the spike added.

11.12 Practical Quanitation Limits (PQL). The PQL is calculated as five to ten times the MDL. See Table #2.

11.13 Method Detection Limit (MDL). A minimum of seven replicate standards are digested and analyzed following 40 CFR Part 136, Appendix B. This is done annually

11.14 Each day, and before any samples can be analyzed, a blank and a high standard are analyzed to set the linear calibration range. At least once every six months the upper limit of the linear dynamic range must be established for each wavelength utilized by determining the signal responses from a minimum of three different concentration standards across the range. The

upper range limit is an observed signal within <±10% of that extrapolated from the lower standards.

11.15 Serial Dilution. One five fold serial dilution must be analyzed for each matrix type and for each waste stream to evaluate the effect of interferences on the non-diluted results. Those elements with concentrations greater than 50 times the MDL will be used to determine if there are any interferences present. A control limit of 10% is used. The client will be informed whenever an element is outside the control limit.

11.16 Each day, and before any samples can be analyzed the polychromater profile is checked. From the main menu select <profile>, select <automatic>, introduce 1000ppm arsenic solution, select <run>. The profile must fall within 0.2 units of zero, if not, select <calc SS>, adjust the vernier dial to the new calculated position.

12.0 INSTRUMENTAL PROCEDURES

12.1 WARNING: Ensure that no pacemaker users are in the vicinity of the spectrometer. RF generator radiation may interfere with pacemaker operation.

A) Prior to initiation of the plasma, the operator should be familiar with the operator's manual supplied by Thermo Jarrell Ash.

B) Before starting the instrument, check the exhaust vents and cooling water to ensure they are operating.

C) Check that the pressure of the argon supply is 50 psi.

D) Check that the drain tube is immersed in at least 2 inches of water in the plastic waste container. Ensure there are no crimps in this tubing.

E) Connect the rinse and sample tubing on the peristaltic pump. Check that there is sufficient rinse solution in the rinse supply container. Start the instrument and allow at least 30 minutes to warm up prior to profile and calibration.

12.2 Sample Evaluation

A) Locate the sample digestates to be analyzed and compare them to the applicable prep log in order to determine the analysis protocols. Check login to determine which elements are required and prepare the appropriate run sequence.

B) Visually inspect the sample digestates, noting which samples were in duplicate and which were spiked. Also note initial and final sample volumes, which laboratory control samples were used and also the sample weights and volumes. Check this against the sample prep log for completeness for all these mentioned items.

C) Samples must be free of particulates before being aspirated. Carefully inspect samples as they are poured into the autosampler tubes. Gravity filter samples as needed.

D) Soil digestates with a final volume of 50 ml must be diluted 5 fold with 2 % nitric acid for matrix matching

13.0 CALCULATIONS

13.1 Calculations are performed by the analytical software with direct reading in ppb (ug/L). The conversion to mg/Kg is as follows:

$$\frac{\text{(Reading in ppb)(sample digestate volume in liters)}}{\text{Sample Wt.in grams X dec. \% solids}} = \text{results in mg/Kg}$$

14.0 ACCEPTANCE CRITERIA

Acceptance criteria can be found in Quality Control Section 11for all QC samples.

15.0 REPORTING OF RESULTS

15.1 Samples are entered into the LIMS system. All appropriate sections must be completed. The data prep page should be printed and put in job folder.

15.2 A batch system is used. Only 20 samples are allowed in a batch. Each batch should have a duplicate sample, a spiked sample, spiked duplicate, a prep-blank and a laboratory control sample. All of these samples should be indicated in the system logbook page.

15.3 Record pH, matrix, initial volume and final volume for each sample.

15.4 Record lot number of all solutions and reagents added.

15.5 Record in comment column if sample is diluted, concentrated, limited, preserved, reprepped or other changes.

16.0 POLLUTION PREVENTION

16.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

16.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

16.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

16.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

16.1.4 Waste Reduction; Reduce the volume of waste generated wherever possible.

16.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

17.0 WASTE MANAGEMENT

17.1 All waste shall be managed in accordance with all state and federal requirements. See the STL-CT Hazardous Waste Management Plan.

17.2 All personnel who handle or generate waste must be trained within six months of employment in proper waste handling and requirements.

17.3 All acid waste is disposed of into a 55 gallon polyethylene drum marked as acid waste.

18.0 SUPPLEMENTAL DOCUMENTS

18.1 **None**

19.0 REFERENCES

19.1 USEPA SW846 Third edition Method No. 6010B.

20.0 SUBSTANTIVE REVISIONS

20.1 Changed name to STL from IEA. Updated method reference

20.2 Added section 3 – Terms and definitions, 16 & 17 for waste management and pollution prevention, renumber other sections: 02/12/00.

20.3 Updated section 6 to include the corporate health and safety SOP: 10/01/2004.

20.4 Updated to Method 6010B, 03/21/05.

20.5 Updated acceptance criteria for ICB and CCB section 11.2 and 11.6, 03/21/05.

20.6 Updated acceptance criteria for MB section 11.7, 03/21/05.

20.7 Updated acceptance criteria for CRI section 11.3, 03/21/05.

20.8 Updated acceptance criteria for PQL section 11.12, 03/21/05.

20.9 Updated linear range section 11.14, 03/21/05.

20.10 Updated section 11.16 to include instrument profile procedure added 17.3 to include disposal

of acid waste; 09/27/07.

Table #1

Quality Control Sample	Control Limit	Failure Action
ICV	± 10 %	Recalibrate
ICB	< 3X MDL	Recalibrate
CRI	± 20 %	Recalibrate
ICSA	± 20 %	Recalibrate
ICSAB	± 20 %	Recalibrate
CCV	± 10 %	Rerun Samples
CCB	< 3X MDL	Rerun Samples
Duplicate	± 20 % RPD	Flag Sample
Sample Spike	± 25 % Recovery	Flag Sample
Prep Blank	< 1/2 Reporting Limit	Reprep Samples
Lab Control Sample	± 20 %, 95 % Confid Win	Reprep Samples

Table #2

Analyte	Wavelength (nm)	PQL Water (ug/L)	PQL Soil (Mg/Kg)
Aluminum	308.20	500.0	258
Antimony	206.83	20.0	11.7
Arsenic	189.00	40.0	8.0
Barium	493.40	5.0	2.0
Beryllium	313.00	5.0	2.0
Cadmium	226.50	10.0	3.0
Calcium	317.93	300.0	85
Chromium	267.70	10.0	3.0
Cobalt	228.61	10.0	2.0
Copper	324.75	10.0	5.0
Iron	271.44	100.0	145
Lead	220.35	10.0	9.0
Magnesium	279.07	100.0	35.0
Manganese	257.61	15.0	2.5
Mercury	253.70	0.4	0.05
Nickel	231.60	10.0	6.25
Potassium	766.49	400.0	200
Selenium	196.02	30.0	16.0
Silver	328.06	6.0	3.0
Sodium	588.90	400.0	94
Thallium	190.80	40.0	22
Vanadium	292.40	6.0	4.0
Zinc	213.85	50.0	20.0
Boron	249.60	60.0	100

Title: SOP for Mercury – Solids, Hot Block Digestion
[Method SW846 7471A]

Approvals (Signature/Date):

Technical Manager Date: 10/17/07	Health & Safety Manager / Coordinator Date: 10-19-07
Quality Assurance Manager Date: 10/17/07	Laboratory Director Date: 10/19/07

This SOP was previously identified as SOP No. MES03205.CT.

Facility Distribution No. _____ Distributed To: _____

Any reference within this document to Severn Trent Laboratories, Inc. or STL, should be understood to refer to TestAmerica Laboratories, Inc. (formerly known as Severn Trent Laboratories, Inc.)

1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 This SOP defines the analysis of samples by cold-vapor atomic absorption for soils, sediments, bottom deposits and sludge–type materials. Samples are digested and then analyzed for mercury by reduction with stannous chloride, which is added in-line by a mercury analyzer.

2.2 The document control number for this document is CT-MES-32, Rev 5.

3.0 TERMS AND DEFINITIONS

3.1 There are many definitions used within the laboratory, which may be generic to all laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used within the laboratory, reference the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 The flameless AA procedure is based on the absorption of radiation at 253.7 nm by mercury vapor. Organic mercury compounds are oxidized; the mercury is reduced to the elemental state, and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of a mercury analyzer. Absorbance (peak height) is measured as a function of mercury concentration and recorded in the usual manner. All samples, calibration standards and quality control samples must be digested prior to analysis.

4.2 This method is based on SW846 Method 7471A.

5.0 INTERFERENCES

5.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide do not interfere with the recovery of added mercury.

5.2 Copper has been reported to interfere; however, copper concentrations as high as 10 mg/L have no effect on mercury recovery.

5.3 High levels of free chlorine interfere with the analysis because chlorine also absorbs at a wavelength of 253.7 nm. Hydroxylamine hydrochloride is added prior to analysis to eliminate chlorine and to reduce the excess permanganate.

5.4 Interference from certain volatile organic compounds that also absorb at 253.7 nm is possible. A preliminary run without reagents should be made if this is suspected.

6.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

6.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

6.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 PPM	Oxidizer Corrosive	0.1 Mg/M3 Ceiling	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation.

in Reagent)	Poison	(Mercury Compounds)	Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact

			may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 Mg/M3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

7.1 SAMPLE COLLECTION: Samples are collected in glass bottles. Sample bottles are not to be reused.

7.2 SAMPLE PRESERVATION: Cool to 4 C.

7.3 HOLDING TIMES: Samples must be analyzed within 28 days of collection. Samples must be analyzed within 26 days of collection if following NYSDEC methodology.

8.0 APPARATUS AND MATERIALS

8.1 Perkin Elmer FIMS 100 Flow Injection Mercury System.

8.2 Environmental Express Hot Block Digestion Unit (SC100)

8.3 Calibrated Thermometer

8.3.1 The temperature of the digestor must be monitored during each batch. This is done by filling a digestion tube ¾ full with reagent water and placing it in a digestion slot. A calibrated thermometer is then placed in the tube and monitored throughout the digestion. Polypropylene digestion tubes with caps (50 ml)

8.4 Eppendorf Pipets (Brinkmann 2000 series, variable, 100uL and 1000uL).

8.4.1 Eppendorf pipets must be calibrated daily at similar volumes used to dispense standards and reagents.

8.4.2 Reagent water stored at room temperature is pipetted into a tared disposable beaker. The milliliters dispensed should equal the weight in grams. Pipets used for standards should be accurate to 0.5% and those used for reagents should be accurate to 1%. If not, calibrate the eppendorf following the manufacturer's instructions. Calibrations are to be recorded in a log specific to each pipet.

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Reagent water - 18 megohm, deionized

9.2 Concentrated Sulfuric Acid, Reagent Grade

9.3 Concentrated Nitric Acid, Trace Grade

9.4 Concentrated Hydrochloric Acid, Trace Grade

9.5 Stannous Chloride (1.1% solution) - Add 11g Tin Chloride Dihydrate (SnCl2.2H2O) to a 1L plastic bottle. Add approximately 700 ml reagent water followed by 30 ml concentrated HCl. Fill to the top with reagent water, cap and mix by inversion. Store this solution at 4 C and discard if it turns green. Always make sure that there is

enough reagent for the entire analytical run. Never mix batches of reagent during a run. However, batches can be combined prior to a run. Prepare fresh daily.

9.6 Sodium chloride-Hydroxylamine hydrochloride solution - Dissolve 120 g of each in 1 L of reagent water.

9.7 Potassium permanganate (5% solution) - Dissolve 125 g of potassium permanganate in 2.5 L of reagent water.

9.8 3% HCl - To approximately 700 ml reagent water add 30 ml concentrated HCl. Add reagent water to make 1L total volume.

9.9 1000 ppm Mercury calibration standard

9.10 Second source Mercury standard (1000 ppm)

9.11 Laboratory Control Standard (ERA metals in soil catolog #540)

9.12 500 ppb intermediate standards for each source – Add 50.0 ul of 1000 ppm mercury to a 100ml volumetric flask containing approximately 70 ml of reagent water and 2 ml of concentrated nitric acid. Add reagent water to the mark, stopper and mix by inverting the flask several times.

9.13 Aqua Regia half strength: To a 250 ml plastic bottle add 80 ml of reagent water followed by 20 ml of concentrated HNO3 and 60 ml of concentrated HCl. Cap and swirl gently to mix. Prepare this solution daily and always in a hood.

10.0 CALIBRATION

10.1 Standards are to be run in ascending order. A five-point linear calibration curve is run and the correlation coefficient must be equal to or greater than 0.995 (i.e., $r \geq 0.995$) before actual samples can be run.

11.0 QUALITY CONTROL

11.1 Method detection limits are calculated according to 40 CFR Appendix B Part 136 and are

performed annually.

11.2 The Practical Quantitation Limit (PQL) for mercury is 0.20 ug/L.

11.3 All stock solutions and standard preparations are logged and coded. All solutions are labeled with the following: analyte, concentration, analyst's initials, date prepared, and expiration date.

11.4 All chemicals will conform to minimum specifications set by the Reagent Chemicals Committee of the American Chemical Society. All chemical inventories are used on a first in first out basis.

11.5 A method blank or prep blank (PB) must be analyzed for every batch of 20 samples or every day, which ever is more frequent. Method blanks are prepared using blank reagent water. All samples with an unacceptable method blank are to be reprepped and rerun.

11.6 One sample duplicate must be analyzed from each batch of 20 or fewer samples. The samples identified as field blanks are not to be used for duplicate analysis.

11.7 One sample spike is performed on every batch of 20 or fewer samples. Samples identified as field blanks may not be used for sample spike analysis.

11.8 One independent standard or laboratory control standard (LCS) for mercury is to be analyzed with each batch of 20 samples. All samples with an unacceptable LCS are to be reprepped and rerun.

11.9 All digestates with concentrations above 10.0 ug/L must be diluted and reanalyzed.

11.10 Initial and Continuing Calibration:

Activity	Frequency
Initial Calibration Verification (ICV)	Immediately after the calibration curve
Initial Calibration Blank (ICB)	Immediately following the ICV
Continuing Calibration Verification (CCV)	After every ten samples, and after the last analytical sample.

Continuing Calibration Blank (CCB)	Immediately following each CCV

If a standard or CCB fails then, the 10 samples prior to that standard must be reanalyzed as well as the samples after that QC.

12.0 SAMPLE PREPARATION and ANALYSIS

12.1 Sample preparation: Weigh approximately 0.6g of sample, using three separate portions, and place in the bottom of a digestion tube. Make sure that very little sample is stuck to the sides of the tube. Add 10 ml half strength aqua regia and heat for 2 minutes in a hot block at 95 C. Cool and add reagent water to the 25 ml mark. Next add 5 ml of potassium permanganate solution and swirl to mix. Samples with high reductive properties will reduce the permanganate and cause the purple color to disappear. Add additional permanganate until the purple color persists. If more than 5 additional ml of permanganate is needed, than a stronger permanganate solution will be needed. Likewise, if after digestion a sample loses its purple color, than it must be redigested using a larger addition of permanganate. Always include an additional reagent blank that contains the same permanganate concentration as the highest sample. Heat for 30 minutes in a hot block maintained at 95 C. Allow samples to cool to room temperature. Add 2 ml of hydroxylamine solution and swirl to mix. Add additional hydroxylamine solution to eliminate the purplish color if needed. Add reagent water accurately to the 50 ml mark, cap and mix the tubes by inverting them several times.

12.2 Standard preparation: Transfer 20, 100, 200, 500 and 1000 ul aliquots of the 500 ppb mercury calibration standard to a series of digestion tubes. This will correspond to 0.2, 1, 2, 5 and 10 ppb mercury in the final diluted digestates. Add reagent water to the 5 ml mark and swirl to mix. Prepare enough calibration verification standards that will be required for the entire run from the second source intermediate, as dictated in section 11.10. Similarly prepare all necessary calibration blanks and laboratory control samples.
To each tube add 5 ml of half strength aqua regia solution and heat for 2 minutes in a hot block at 95 C. Cool and add reagent water to the 25 ml mark. Next add 5 ml of potassium permanganate solution and swirl to mix. Heat for 30 minutes in a hot block maintained at 95 C. Allow samples to cool to room temperature. Add 2 ml of hydroxylamine solution and swirl to mix. Add additional hydroxylamine solution to eliminate the purplish color if needed. Add reagent water accurately to the 50 ml mark, cap and mix the tubes by inverting them several times.
Note: Always make sure that there is enough of all the reagents that will be needed for the entire batch of digestions. Reagent batches can only be combined prior to sample preparation.

12.3 Instrument Operation

A) Turn on instrument and printer.

B) Open FIMS Folder, open AA winlab analyst folder.

C) On workspace screen open automated analysis.

D) Open sample information and fill in all sample criteria.

E) Open File and Save As using the date the samples are being analyzed.

F) Print sample info file and close widow.

G) Open Automated analysis window, select browse, open file to be analyzed and click OK.

H) Click "use entire sample" box.

I) Click method STLHG at top of screen and click OK

J) Open FIAS window and click pump 1, let run for two minutes, shut off pump 1 and close window.

K) In automated analysis window select "analyze all" to start analysis.

L) Run Sequence proceeds as follows:

Standards in ascending order.

ICV, ICB

10 samples (includes prep blanks, duplicates, and spikes).

CCV, CCB

Repeat the above two steps until all samples are analyzed.

12.2 Digestates that exceed 10 ppb mercury must be diluted with dilution solution and reanalyzed.

12.3 Dilution solution: To a 1L plastic bottle add approximately 500 ml of reagent water followed sequentially by 12.5 ml of concentrated nitric acid, 37.5 ml of concentrated hydrochloric acid, 100 ml of 5% permanganate solution and 40 ml of hydroxylamine solution, mixing after each addition. Fill the bottle completely with reagent water, cap and mix by inversion.

12.4 Digestates must be free of particulates before being analyzed. Therefore, filtering and or diluting must be employed to limit particulates.

13.0 CALCULATIONS

13.1 A linear calibration curve is used to calculate results.

13.2 Sample Spike Recovery: $\% R = [(SSR - SR)/SA] \times 100$

SSR is Sample spike recovery in ug/L
SR is Sample result in ug/L
SA is Spike added in ug/L

13.3 $\% RPD = \{(| A - B |)/ [(A + B)/2]\} \times 100$

A is the sample result in ug/L
B is the duplicate result in ug/L

13.4 All data is downloaded to the labnet data system. Open C: find the data file to be downloaded and drag file to the L: directory. Open L: find the data to be downloaded and drag file to MERC1. Data is now ready to be imported for reporting.

14.0 ACCEPTANCE CRITERIA

Initial Calibration Verification ± 10%
Continuing Calibration Verification ± 20%
Initial Calibration Blank: (<MDL)
Method Blank: (< ½ reporting limit)
Duplicates: ± 20%
± PQL if sample concentration < 5 x Reporting Limit
Spikes: ± 25%
Laboratory Control Sample: within manufacturers control limits.

15.0 REPORTING OF RESULTS

Practical Quantitation Limits: 0.20
Units of measure: Mg/Kg
Significant Figures: 3 figures

16.0 POLLUTION PREVENTION

16.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

16.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

16.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

16.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

16.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

16.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

17.0 WASTE MANAGEMENT

17.1.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

17.1.2 All acid waste is disposed into a 55-gallon plastic drum located in the satellite accumulation area.

18.0 SUPPLEMENTAL DOCUMENTS

18.1 None

19.0 REFERENCES

19.1 USEPA SW846 Test Methods for Evaluating Solid and Hazardous Wastes, Third Edition, method 7471, including revisions.

19.2 Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82-055 December 1982, Method 245.1.

20.0 SUBSTANTIVE REVISIONS

20.1 Used daily working standard preparation from section 9 to section 12. Revised entire section 12 to include instrument procedures for the PE FIMS Mercury analyzer, added dilution solution to section 9, expanded QC sections 11.5,11.8, 11.10; 4/28/03 MKC.

20.2 Updated section 6 to include the corporate health and safety SOP January 28, 2004.

20.3 Modified section 12.1 to use 10ml aqua regia, 03/18/2005.

20.4 Modified section 14 and Table 1 to meet method requirements, 03/18/2005.

20.5 Updated sections 10 and 13 to include linear calibration calculation, Added new TestAmerica

SOP header and control number; 09/27/07.

Table #1

Quality Control Sample	Control Limit	Failure Action
ICV	± 10 %	Recalibrate
ICB	< MDL	Recalibrate
CCV	± 20 %	Rerun Samples
CCB	< ½ reporting limit	Rerun Samples
Duplicate	± 20 % RPD	Flag Sample
Sample Spike	± 25 % Recovery	Flag Sample
Prep Blank	< ½ reporting limit	Reprep Samples

Title: SOP for Mercury - Aqueous, Hot Block Digestion
[Method SW846 7470A]

Approvals (Signature/Date):

Technical Manager	Date 10/18/07	Health & Safety Manager / Coordinator	Date 10-19-07
Quality Assurance Manager	Date 10/17/07	Laboratory Director	Date 10/19/07

This SOP was previously identified as SOP No. MES03107.CT.

Facility Distribution No. _____ Distributed To:_____

1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 This SOP defines the analysis of samples by cold-vapor atomic absorption for samples of an aqueous matrix. Samples are digested and then analyzed for mercury by reduction with stannous chloride, which is added in-line by a mercury analyzer.

2.2 The element determined is defined as "Total Mercury" for an unfiltered sample and as "Dissolved Mercury" for a filtered sample. All samples, calibration standards and quality control samples must be digested prior to analysis.

2.3 The document control number for this document – CT-MES-31, Rev 7.

3.0 TERMS AND DEFINITIONS

3.1 There are many definitions used within the laboratory, which may be generic to all laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used within the laboratory, reference the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 The flameless AA procedure is based on the absorption of radiation at 253.7 nm by mercury vapor. Organic mercury compounds are oxidized, additional potassium permanganate will be added to ensure that the purple color persists for at least 15 minutes; the mercury is reduced to the elemental state, and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of a mercury analyzer. Absorbance (peak height) is measured as a function of mercury concentration and recorded in the usual manner.

4.2 This method is based on SW846 Method 7470A and EPA Method 245.1.

5.0 INTERFERENCES

5.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide do not interfere with the recovery of added mercury.

5.2 Copper has been reported to interfere; however, copper concentrations as high as 10 mg/L have no effect on mercury recovery. Samples suspected of containing appreciably greater amounts must be diluted prior to digestion.

5.3 High levels of free chlorine interfere with the analysis because chlorine also absorbs at a wavelength of 253.7 nm. Hydroxylamine hydrochloride is added prior to analysis to eliminate chlorine and to reduce the excess permanganate.

5.4 Interference from certain volatile organic compounds that also absorb at 253.7 nm is possible. A preliminary run without reagents should be made if this is suspected.

6.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

6.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

6.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury	Oxidizer	0.1 Mg/M3	Extremely toxic. Causes irritation to the

(1,000 PPM in Reagent)	Corrosive Poison	Ceiling (Mercury Compounds)	respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact

			may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.

Potassium Permanganate	Oxidizer	5 Mg/M3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

7.1 SAMPLE COLLECTION: Samples are collected in 500-mL bottles. Sample bottles are not to be reused.

7.2 SAMPLE PRESERVATION: Sample preservation is with nitric acid and preserved to a pH of less than two.

7.3 HOLDING TIMES: Samples must be analyzed within 28 days of collection.

8.0 APPARATUS AND MATERIALS

8.1 Perkin Elmer FIMS 100 Flow Injection Mercury System.

8.2 Hot Block Digestion Unit (SC100)

8.3 Thermometer - glass, mercury, 1 degree increments, 100 degree calibrated thermometer.

8.3.1 The temperature of the digestor must be monitored during each batch. This is done by filling a digestion tube ¾ full with reagent water and placing it in a digestion slot. A calibrated thermometer is then placed in the tube and monitored throughout the digestion.

8.4 Polypropylene digestion tubes with caps (50 ml).

8.5 Eppendorf Pipets (Brinkmann 2000 series, variable, 100uL and 1000uL).

8.5.1 Eppendorf pipets must be calibrated daily at similar volumes used to dispense standards and reagents.

8.5.2 Reagent water stored at room temperature is pipetted into a tared disposable beaker. The milliliters dispensed should equal the weight in grams. Pipets used for standards should be accurate to 0.5% and those used for reagents should be accurate to 1%. If not, calibrate the eppendorf following the manufacturer's instructions. Calibrations are to be recorded in a log specific to each pipet.

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Reagent water - 18 megohm, deionized

9.2 Concentrated Sulfuric Acid, Reagent Grade

9.3 Concentrated Nitric Acid, Trace Grade

9.4 Concentrated Hydrochloric Acid, Trace Grade

9.5 Stannous Chloride (1.1% solution) - Add 11g Tin Chloride Dihydrate (SnCl2.2H2O) to a 1L plastic bottle. Add approximately 700 ml reagent water followed by 30 ml concentrated HCl. Fill to the top with reagent water, cap and mix by inversion. Store this solution at 4 C and discard if it turns green. Always make sure that there is enough reagent for the entire analytical run. Never mix batches of reagent during a run. However, batches can be combined prior to a run.

9.6 Sodium chloride-Hydroxylamine hydrochloride solution - Dissolve 120 g of each in 1 L of

reagent water.

9.7 Potassium permanganate (5% solution) - Dissolve 125 g of potassium permanganate in 2.5 L of reagent water.

9.8 Potassium persulfate (5% solution) - Dissolve 125 g of potassium persulfate in 2.5L of reagent water.

9.9 3% HCl - To approximately 700 ml reagent water add 30 ml concentrated HCl. Add reagent water to make 1L total volume.

9.10 1000 ppm Mercury calibration standard

9.11 Second source Mercury standard (1000 ppm)

9.12 Laboratory Control Standard (ERA or equivalent)

9.13 500 ppb intermediate standards for each source – Add 50.0 ul of 1000 ppm mercury to a 100ml volumetric flask containing approximately 70 ml of reagent water and 2 ml of concentrated nitric acid. Add reagent water to the mark, stopper and mix by inverting the flask several times.

9.14 Dilution solution: To a 1L plastic bottle add approximately 500 ml of reagent water followed sequentially by 25 ml of concentrated sulfuric acid, 12 ml of concentrated nitric acid, 80 ml of 5% permanganate solution, 40 ml of 5% persulfate solution and 30 ml of hydroxylamine solution, mixing after each addition. Fill the bottle completely with reagent water, cap and mix by inversion.

10.0 CALIBRATION

10.1 Standards are to be run in ascending order. A five-point linear calibration curve is run and the correlation coefficient must be equal to or greater than 0.995 (i.e., $r \geq 0.995$) before actual samples can be run.

11.0 QUALITY CONTROL

11.1 Method detection limits are calculated according to 40 CFR Appendix B Part 136 and are performed annually.

11.2 The Practical Quantitation Limit (PQL) for mercury is 0.20 ug/L.

11.3 All stock solutions and standard preparations are logged and coded. All solutions are labeled with the following: analyte, concentration, analyst's initials, date prepared, and expiration date.

11.4 All chemicals will conform to minimum specifications set by the Reagent Chemicals Committee of the American Chemical Society. All chemical inventories are used on a first in first out basis.

11.5 A method blank or prep blank (PB) must be analyzed for every batch of 20 samples or every day, which ever is more frequent. Method blanks are prepared using blank reagent water. All samples with an unacceptable method blank are to be reprepped and rerun.

11.6 One sample duplicate must be analyzed from each batch of 20 or fewer samples. The samples identified as field blanks are not to be used for duplicate analysis.

11.7 One sample spike is performed on every batch of 20 or fewer samples. Samples identified as field blanks may not be used for sample spike analysis.

11.8 One independent standard or laboratory control standard (LCS) for mercury is to be analyzed with each batch of 20 samples. ERA's mercury standard catalog #666, or the LCS can be prepared from the same source standard as the initial calibration standard. All samples with an unacceptable LCS are to be reprepped and rerun.

11.9 All digestates with concentrations above 10.0 ug/L must be diluted and reanalyzed.

11.10 Initial and Continuing Calibration:

Activity	Frequency
Initial Calibration Verification (ICV)	Immediately after the calibration curve
Initial Calibration Blank (ICB)	Immediately following the ICV
Continuing Calibration Verification (CCV)	After every ten samples, and after the last analytical sample.
Continuing Calibration Blank (CCB)	Immediately following each CCV

If a standard or CCB fails then, the 10 samples prior to that standard must be reanalyzed as well as the samples after that QC.

12.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

12.1 Sample preparation: Pour a well shaken sample into a 50 ml digestion tube accurately to the 25 ml mark. For dilutions, pipet the appropriate amount of well shaken sample to the digestion tube and add reagent water accurately to the 25 ml mark. Add 1.25 ml concentrated sulfuric acid and 0.6 ml concentrated nitric acid to each tube, mixing between additions. Add 4.0 ml of potassium permanganate solution to each tube and let stand for at least 15 minutes. Samples with high reductive properties will reduce the permanganate and cause the purple color to disappear. Add additional permanganate until the purple color persists. If more than 5 additional ml of permanganate is needed, than a stronger permanganate solution will be needed. Likewise, if after digestion a sample loses its purple color, than it must be redigested using a larger addition of permanganate. Always include an additional reagent blank that contains the same permanganate concentration as the highest sample. Add 2.0 ml of potassium persulfate to each tube and heat for two hours in a hot block maintained at 95 C. Allow samples to cool to room temperature. Add 1.5 ml of hydroxylamine solution and swirl to mix. Add additional hydroxylamine solution to eliminate the purplish color if needed. Add reagent water accurately to the 50 ml mark, cap and mix the tubes by inverting them several times.

12.2 Standard preparation: Transfer 20, 100, 200, 500 and 1000 ul aliquots of the 500 ppb mercury calibration standard to a series of digestion tubes. This will correspond to 0.2, 1, 2, 5 and 10 ppb mercury in the final diluted digestates. Add reagent water to the 25 ml mark and swirl to mix. Prepare enough calibration verification standards that will be required for the entire run from the second source intermediate, as dictated in section 11.10. Similarly prepare all necessary calibration blanks and laboratory control samples.
To each tube add 1.25 ml of concentrated sulfuric acid and 0.6 ml of concentrated nitric acid, mixing after each addition. Add 4.0 ml of permanganate solution and allow to stand for at least 15 minutes. Add 2.0 ml of potassium persulfate solution to each tube and heat in a hot block maintained at 95 C. Allow samples to cool to room temperature. Add 1.5 ml of hydroxylamine solution and swirl to mix. Add additional hydroxylamine solution to eliminate the purplish color if needed. Add reagent water accurately to the 50 ml mark, cap and mix the tubes by inverting them several times.
Note: Always make sure that there is enough of all the reagents that will be needed for the entire batch of digestions. Reagent batches can only be combined prior to sample

preparation.

12.3 Instrument Operation

A) Turn on instrument and printer.

B) Open FIMS Folder, open AA winlab analyst folder.

C) On workspace screen open automated analysis.

D) Open sample information and fill in all sample criteria.

E) Open File and Save As using the date the samples are being analyzed.

F) Print sample info file and close widow.

G) Open Automated analysis window, select browse, open file to be analyzed and click OK.

H) Click "use entire sample" box.

I) Click method STLHG at top of screen and click OK

J) Open FIAS window and click pump 1, let run for two minutes, shut off pump 1 and close window.

K) In automated analysis window select "analyze all" to start analysis.

L) Run Sequence proceeds as follows:

Standards in ascending order.

ICV, ICB

10 samples (includes prep blanks, duplicates, and spikes).

CCV, CCB

Repeat the above two steps until all samples are analyzed.

12.5 Digestates that exceed 10 ppb mercury must be diluted with dilution solution and reanalyzed.

12.6 Digestates must be free of particulates before being analyzed. Therefore, filtering and or diluting must be employed to limit particulates.

13.0 CALCULATIONS

13.1 A linear calibration curve is used to calculate results.

13.2 Sample Spike Recovery: $\% R = [(SSR - SR)/SA] \times 100$

SSR is Sample spike recovery in ug/L
SR is Sample result in ug/L
SA is Spike added in ug/L

13.3 $\% RPD = \{(| A - B |)/ [(A + B)/2]\} \times 100$

A is the sample result in ug/L
B is the duplicate result in ug/L

14.0 ACCEPTANCE CRITERIA

Initial Calibration Verification $\pm$ 10%
Continuing Calibration Verification $\pm$ 20%
Initial Calibration Blank: (<MDL)
Method Blank: (< ½ reporting limit)

Duplicates	$\pm$ 20%
	$\pm$ PQL if sample concentration < 5 x Reporting Limit
Spikes	$\pm$ 25%
Laboratory Control Sample:	$\pm$ 20%

15.0 REPORTING OF RESULTS

Practical Quantitation Limits: 0.20	
Units of measure:	ug/L
Significant Figures:	3 figures

16.0 POLLUTION PREVENTION

16.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

16.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

16.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

16.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

16.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

16.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

17.0 WASTE MANAGEMENT

17.1.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

17.1.2 All acid waste is disposed into a 55-gallon plastic drum located in the satellite accumulation area.

18.0 SUPPLEMENTAL DOCUMENTS

18.1 None

19.0 REFERENCES

19.1 USEPA SW846 Test Methods for Evaluating Solid and Hazardous Wastes, Third Edition, method 7470, including revisions.

19.2 Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82-055; December 1982, Method 245.1.

20.0 SUBSTANTIVE REVISIONS

20.1 Used daily working standard preparation from section 9 to section 12. Revised entire section 12 to include instrument procedures for the PE FIMS Mercury analyzer, added dilution solution to section 9, expanded QC sections 11.5, 11.8, 11.10; 4/28/03 MKC.

20.2 Updated section 6 to include the corporate health and safety SOP January 28, 2004.

20.3 Updated section 7 to remove glass and plastic bottles. Updated holding time to 28 days form time of collection.

20.4 Updated sections 10 and 13 to include linear calibration calculation, Added new TestAmerica SOP header and control number; 09/27/07.

Table #1

Quality Control Sample	Control Limit	Failure Action
ICV	± 10 %	Recalibrate
ICB	< MDL	Recalibrate
CCV	± 20 %	Rerun Samples
CCB	< ½ reporting limit	Rerun Samples
Duplicate	± 20 % RPD	Flag Sample
Sample Spike	± 25 % Recovery	Flag Sample
Prep Blank	< ½ reporting limit	Reprep Samples

Title: SOP for GC/MS Volatiles

[Method SW846 8260B]

Approvals (Signature/Date):

Technical Manager	Date: 1-18-08	Health & Safety Manager / Coordinator	Date
Quality Assurance Manager	Date: 1-18-08	Laboratory Director	Date: 1-18-08

This SOP was previously identified as SOP No. MSS02808.CT.

Facility Distribution No. _____ **Distributed To:_____**

1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 The objective of this document is to outline the techniques for determining the presence and concentration of various volatile organic target and non-target compounds in multi-media, multi-concentration samples. The 8260B compounds for this method are listed in Table 1.0. Table 2.0 lists the expanded Appendix IX target compounds which are applicable to this method. The extraction method used in this procedure is purge and trap which is coupled with a gas chromatograph/mass spectrometer analysis.

2.2 It is the policy of TESTAMERICA and of the GC/MS Group to ensure that we administer contracts and orders for goods and services in a manner that is fully compliant with governmental laws and regulations, as well as the TESTAMERICA Policy Statement on Business Ethics and Conduct.

2.3 The document control number for this SOP is CT-MSS-28, Rev 7.

3.0 Terms and Definitions

3.1 There are many definitions used within the laboratory, which may be generic to all Laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used within the laboratory, reference the SOP of Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 This method employs the technique of purge and trap, coupled with a gas chromatograph/mass spectrometer analysis. An aliquot of sample, usually 5 ml or 25 ml of water, 5 g of soil for low level soil method, and 5 g of soil collected with methanol and extracted for medium level soil method, is purged in a gas tight chamber with UHP grade helium to remove the volatile compounds. The vapor is swept through a sorbent column where the volatiles are trapped. Next the sorbent trap is heated and back flushed, thereby desorbing the volatiles onto the analytical column within the gas chromatograph. The fused silica capillary column is then temperature programmed to separate the volatiles prior to detection by the mass spectrometer.

4.2 This SOP is based on USEPA SW846 5030B/5035/8260B Methods.

4.3 The following deviations from the method are noted:

. The selected internal standards for this 8260B Method are:

- Fluorobenzene
- 1,4-dichlorobenzene-d4
- Chlorobenzene-d_5

. The selected surrogates for this 8260B Method are:

- Dibromofluoromethane
- 1,2-Dichloroethane-d_4
- Toluene-d_8
- Bromofluorobenzene

. The routine 8260B target list is the TCL list per Table 1.0.

. Method 8260B can be either 5 ml or 25 ml purge per methodology. This must be specified by the client prior to sample analysis. In many cases the detection limits requested can be achieved by 5 ml sample volume using the Agilent 5973 with a lower calibration range.

5.0 INTERFERENCES

5.1 Method interferences may be caused by contaminants in solvents, reagents, out-gassing from equipment plumbing, and laboratory solvent vapors. This can lead to discrete artifacts and/or elevated baseline in the gas chromatograph. All these materials must be demonstrated to be free from interferences by the running of laboratory reagent blanks.

5.2 Interferences may also be caused by the diffusion of volatiles through the septum seal during storage and handling. A holding blank prepared from reagent water is stored with the samples and analyzed to serve as a check. Holding blanks are prepared weekly for each volatile sample storage unit. The results for the holding blank are reported by GC/MS and submitted to the QA/QC Manager to be filed for future reference.

5.3 Only Approved lots of Purge and Trap grade methanol shall be used for standards and sample dilutions.

5.4 Only reagent water free from Volatile organics can be used in the volatiles laboratory for sample dilutions, reagent blanks, and aqueous standards.

6.0 SAFETY

SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in both the Gas Chromatograph and Mass Spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.

6.1 Analysts shall treat all samples as if they are hazardous and take all appropriate safety precautions. Analysts shall wear, if needed:

- lab coats
- safety glasses with side shields and
- chemical resistant gloves

when handling samples or preparing standards.

6.2 Solvents and all standards shall be used in the fume hoods to minimize environmental exposure to solvent vapors.

6.3 Material Safety Data Sheets for all chemicals used in the operation are present in the laboratory for immediate access.

7.0 SAMPLE PRESERVATION AND STORAGE

7.1 All samples for volatile analysis must be protected from light and refrigerated at 4□C from the time of receipt until analysis.

7.2 All HCL preserved samples for volatile analysis shall be analyzed within 14 days of sample collection, or seven days unpreserved.(the laboratory must be notified if samples are unpreserved to ensure unpreserved samples can be run within holding time) NYSDEC 8260B samples must be analyzed within ten days of receipt.

7.3 Refer to the sample control processing, sample removal, and log-in SOP's in section 15.0.

7.4 Low level soil analysis will be preserved with reagent grade Sodium bisulfate or Reagent water will be used if the sample effervesces when it comes in contact with the Sodium Bisulfate solution. If the soil is place in water, this sample will be run that day, or frozen at a slight 30-45 degree angle to prevent breakage, for up to fourteen days. Client must specify at time of bottle order if no preservative is required. Encore containers or equivalent may be used for low level soil analysis. If these are used the soil plug must be transferred within 48 hours from collection.

7.5 Methanol preserved vials will be sent out for projects requesting the high concentration soil analysis. Encore containers or equivalent may be used for medium level soils. If these are used the soil plug must be transferred into methanol within 48 hours from collection. A 14 day hold from collection is used for methanol. Client must also specify what volume of methanol is required, if surrogates need to be pre-spiked prior to being collected in the field, and what soil volume will be sampled. Laboratory default is 10mls of un-spiked methanol to accept 5 grams of soil in the field.

8.0 APPARATUS AND MATERIALS

8.1 Purge and trap concentrator : Encon

8.2 Purge and trap autosampler - or Archon multiple position autosampler

8.3 Volatile Trap - VOACARB 3000 traps packed with: Carbopack B/Carboxen 1000 and 1001 .

8.4 GC/MS/DS System

8.4.1 Hewlett-Packard Model, 5975, 5973, 5972, 5971 or 5970 GC/MS with jet separator, or direct capillary interface, capable of scanning from 35 to 300 amu every two seconds or less, utilizing 70 volts (nominal) electron energy in the EI ionization mode, and producing a mass spectrum which meets all the instrument performance criteria when 50 ng of BFB is injected through the GC inlet. Refer to Table 3.0 for the performance criteria. and section 12.1.1 for the instrumental conditions.

8.4.2 Direct interface, or split injection port from purge and trap transfer line to GC column

8.4.3 GC Column – Restek RTX-VMS 20 meter 0.18 mm ID 1.00 micron film thickness or 75 m x 0.53 mm ID x 3.0 um film thickness Supelco 624 fused silica widebore capillary column, or equivalent narrow bore such as 0.18 or 0.25 are used with split injection ports.

8.4.4 Chemstation / Target software capable of continuous acquisition and storage, on machine readable media, of all mass spectra obtained throughout the duration of the chromatographic

program. The computer has software that allows searching any data file for ions of a specified mass and plotting such ion abundances versus time or scan number (EICP). Software also allows integrating the abundance in any EICP between specified time limits. Also, software allows for the comparison of sample non-target spectrum against reference library spectra. The most recent release of the NIST/EPA/MSDC mass spectral library shall be used as the reference library. The data system flags all manual edits with "M" qualifier.

8.4.5 Target network tape backup system.

8.4.6 Syringes – Gas-tight microsyringes 25 ul and larger, 0.006 inch ID needle; 5 ml gas-tight syringes with shut off valve, all syringes used shall be gas-tight

8.4.7 Balance – top loading balance capable of weighing +/-0.1 g, and an analytical balance capable of weighing +/-0.0001 grams. This balance must be checked for calibration once, prior to use with NIST weights. The range will be from 1gram to 100grams to bracket the working range.

8.4.8 Methanol – Purge and Trap Grade

8.4.9 Fritted sparger , culture tube, or 40ml Voa vial.

8.4.10 5ml syringe –with Luer ends, if applicable to the purging device

8.4.11 Glassware

- Bottle – 15 ml, screw cap, with Teflon cap liner
- Volumetric flasks – class A with ground glass stoppers
- Vials – various for standards

8.4.12 A heater or heated water bath capable of maintaining the purge device at 40□C +/- 1□C attached to a stir plate or sonicator that will agitate the low level soil sample, but not for waters or medium level soils

8.4.13 pH paper – narrow range (0-6pH units) and wide (0-14pH units)- All aqueous samples will have a pH taken and recorded in the injection logbook. Any pH readings above 2 will be addressed in a corrective action, if samples are analyzed more than seven days from collection.

8.4.14 Magnetic stir bars

8.4.15 Polyethylene glycol (PEG)

9.0 REAGENTS AND STANDARD PREPARATION

Refer to the volatiles standards preparation SOP, referenced in section 15.0, for details of standard preparation.

9.1 Reagent Water - Laboratory certified water, free from contaminants.

9.2 Dilution Methanol - Approved lots of Purge and Trap Grade or equivalent.

9.3 Stock Standards - certified standards purchased from commercial sources containing ampulated mixes of target compounds, matrix spike compounds, surrogates, and internal standards are used by the laboratory as stock standards. New ampules are opened every few months, or sooner, if the standard has degraded, evaporated, or the manufactures' date has expired. Ampules containing Gases and Ketones may need to be opened more often.

9.4 Working Standards

9.4.1 Instrument performance check solution - 4-Bromofluorobenzene (BFB) 25 ng/ul solution of BFB in methanol is prepared every six months, or sooner if the solution has degraded or evaporated. Add 2 ul of this solution into 5ml of reagent water for a 50ng concentration. Or can be directly injected into the injection port.

Compound Standard	Initial Concentration	Amount Used	Final Volume	Final Concentration
4-Bromofluorobenzene	25,000ppm	5 ul	5 mL	25 ppm

9.4.2 Calibration Standard Solution

A 50ppm working calibration standard containing all the volatile target compounds in methanol is prepared monthly, or sooner, if the solution has degraded or evaporated. (125ppm or other levels can be made at the discretion of the analyst. The attached chart is presented as an example)

Full Cal std	Initial concentration	Amount added in ul's	Final Volume	Final concentration
Custom Supelco Mix 2026610	2000ppm	625 ul	25 mL	50ppm
502/524 Supelco Vol organic				
Mix 6 supelco				
Chloroprene supelco				
Ketones supelco				
High Concentration in house supelco / ultra	10,000ppm (varied)	125 ul		

High Concentration	Initial concentration	Amount added in ul's	Final Volume	Final concentration
Acrolein	Neat	59.6 ul	1 mL	50,000 ppm
2-Chloroethylvinylether		9.5 ul		10,000 ppm
Vinyl Acetate		10.7 ul		10,000 ppm
1,4-Dioxane		96 ul		100,000 ppm
Acetonitrile		140 ul		100,000 ppm
Isobutanol		124 ul		100,000 ppm

9.4.3 Internal Standard (IS) Spiking Solution

A 125 ppm IS spiking solution containing fluorobenzene, chlorobenzene-d_5, and 1,4-dichlorobenzene-d4 in methanol is prepared as needed, unless the solution has degraded or evaporated. One microliter (ul) is automatically added by the Archon to give a concentration of 25 ug/L.

Compound **Standard**	Initial **Concentration**	Amount **Used**	Final **Volume**	Final **Concentration**
Internal Standard	2500ppm	250 ul	5 mL	125 ppm

9.4.4 System Monitoring Compound (SMC) Spiking Solution

A 125 ppm SMC spiking solution containing Dibromofluoromethane, 1,2-dichloroethane-d_4, toluene-d_8, and 4-bromofluorobenzene in methanol is prepared weekly, or sooner, if the solution has degraded or evaporated. One microliter (ul) is automatically added by the Archon to give a concentration of 25 ug/L.

Compound Standard	Initial Concentration	Amount Used	Final Volume	Final Concentration
Surrogate Standard	2500ppm	250 ul	5 mL	125 ppm

9.4.5 Full Matrix Spiking (MS) Solution

A 50 ppm FMS solution containing the target compounds. (same solution as calibration standard see section 9.2.2).

9.4.6 LCS Spiking Solution (LCS)(20ppb_QCS)

A 25 ppm LCS solution containing all TCL, along with other compounds is prepared as needed from a source independent of the calibration standard in methanol.

LCS spiking standard	Initial concentration	Amount added in ul's	Final Volume	Final concentration
Custom cal Mix Restek	2000-40,000ppm	62.5 ul	5 ML	25ppm
502.2 2000mega mix Restek	2000ppm	62.5 ul		
Voa Cal mix 1 Restek	5000ppm	25 ul		
502.2 Cal Mix Restek	2000ppm	62.5 ul		

10.0 CALIBRATION

10.1 Calibration Standards

Five or (six point) aqueous initial calibration standards containing all the volatile target compounds and SMC's are prepared at the 5, 20, 50, 100, and 200 ug/L levels.

(0.5,2,5,20,50,100 ug/l on Agilent 5973 GC/MS) These standards are prepared from working standards in section 9.0.

The methanol purged in the aqueous calibration standards must not exceed 1% by volume.

10.2 The working calibration of this method is defined by the initial calibration curve, 5 ug/L to 200 ug/L. All samples with target compounds exceeding the upper curve point must be diluted to within the upper half of the calibration range. No compounds can be over the Practical Quantitation Limit (PQL) for that particular compound. Archon / Encon auto-samplers / Concentrators must be deemed free of carry-over prior to continuing sample analysis.

10.3 Calibration curve preparation

The calibration curve is prepared by adding the following amounts of working calibration standard and SMC are added to 5 ml, or of reagent water, achieving the indicated concentrations: Examples as follows.

5 ml Purge

Conc. Level	Volume to Add
5 ug/L	1 uL
20 ug/L	4 uL
50 ug/L	10 uL
100 ug/L	20 uL
200 ug/L	40 uL

For 5ml low level calibration the following example can be used.

In a 100mL volumetric brought to volume with reagent water minus 240uls, add 80 ul of 125ppm calibration check solution. Then add 80ul of 125ppm surrogate solution. This will be the working solution for all calibration points prepared in 44ml volatile vials for use on the Archon. Alternate methods of preparing the standard can be done by using methanolic standards and micro syringes. All bench notes that are used for calculations must be given a document control number from the Quality control department prior to posting.

Example:

Curve point ug/L	Amount of reagent water in voa vial	Amount of working standard from volumetic
0.5	43.6 mls	220 ul
2	43.1 mls	880 ul
5	41.8 mls	2200 ul
20	35.2 mls	8.8 mls
50	22 mls	22 mls
100	0 mls	44 mls

One microliter (ul) of internal standard is automatically added by the Archon to give a concentration of 25 ug/l to all calibration standards for a final concentration of 25 ug/L. For the 25ml purge the IS spiking solution is at 5ug/l.

An initial calibration must be analyzed on each GC/MS system upon column installation, source cleaning, or whenever corrective action is taken which may affect the initial calibration criteria, or if the continuing calibration criteria can not be met.

Separate initial and continuing calibration must be analyzed for water samples, and low level soil samples (unheated versus heated/agitated purge). Extracts of medium level soil samples may be analyzed using the calibrations for water.

Quantitation is based on the average response factor from the initial calibration.

10.4 Acceptance criteria for the Initial Calibration

The initial calibration criteria must meet the following:

- Calibration Check Compounds (CCC) must have RF's whose percent relative standard deviations are less than 30 percent.

- System Performance Check Compounds (SPCC) Average RF must be equal to or greater than 0.100 except for chlorobenzene and 1,1,2,2- tetrachloroethane must be 0.30 or higher.

10.4.1 The percent relative standard deviation (%RSD) should be less than 15 % for each compound of interest.

1. If the %RSD is less than 15 % then the average RF from the initial calibration curve is used for quantitation of the compound.

2. If the **mean** %RSD is below 15% for **all** compound in the initial calibration, then average RF can be used, if not, then linear regression is used for quantitation for those compounds above 15%. The minimum correlation coefficient for any compound using linear regression must also be 0.99. A copy of the linear regression plot from the calibration curve shall be included in the raw data. Samples can be run in the same initial calibration 12 hour BFB clock, without running a calibration check standard just an acceptable method blank and QCS is required.

3. There will be other clients that request particular criteria that is above and beyond the scope of this S.O.P. In these cases the client's criteria must be taken into consideration. Their criteria may enhance the basic requirements of this S.O.P. However; all criteria described in this S.O.P. must be followed. Additional client requests must be agreed upon prior to sample analysis.

   See Appendix B for any custom client requirements. Based on this S.O.P. with client modifications.

   See Appendix B for USACOE differences and requirements for calibration

10.5 Continuing calibration preparation

The continuing calibration standard is the 50 ug/L for 5971 instruments, and 20 ug/L for 5973 instruments. This standard can be part of the initial calibration or is run as a separate standard prepared as above at 50 ug/L / 20 ug/L.

The continuing calibration standard must be analyzed every 12 hours to verify that the initial calibration is still valid. Periodically the continuing calibration level should vary in concentration as a system check standard to satisfy NELAC requirements. This should be done during a 12 hour calibration, but not used as the continuing calibration for the associated samples.

If a continuing calibration fails for any reason, note the reason for failure in the comments' section of the logbook. After two attempts the analyst must determine if an initial calibration needs to be analyzed, or if an educated decision can be made to meet the continuing calibration with the analysis of further calibration checks. One steadfast rule is the last calibration check that is analyzed must be the one used.

Note: Method 8000 and TESTAMERICA policy recommends running an initial calibration after two calibration checks. Currently analysts will note reasons for failures in the comment section of the logbook. After running two calibration checks, the instrument shall be re-tuned, if in the expert opinion of the analyst that a calibration curve is not

needed, but either maintenance, or standard preparation will allow for acceptable continuing calibration verification, then this procedure will be acceptable.

10.6 Continuing Calibration Acceptance criteria

The internal standard areas are compared to the mid-point of the initial calibration standard. If the internal standard areas are within 50-100% Difference and the requirements below are met then the calibration check is successful.

- Calibration Check Compounds (CCC) - percent RSD 20 percent maximum
- System Performance Check Compounds (SPCC) - 0.1minimum RF (0.3 for Chlorobenzene and 1,1,2,2-Tetrachloroethane)

All other compounds shall be reviewed for accuracy. Various clients request different criteria to be set for non-CCC compounds. This may be present is special instruction at login, a QAPP, or Review Appendix B for client specific requirements.

11.0 QUALITY CONTROL

11.1 Method detection limit determination is required by this method annually. A quarterly MDL check at 0.5ppb will be run on each instrument running 8260LL (low method) or 2ppb on instruments running 8260. Soil MDL's are run with a heated purge. Both water and medium level soil MDL's are run without a heated purge.

11.2 Quantitation limits or practical quantitation limits (PQL) for this method are defined by three to five times the method detection limit for each compound. PQL's will vary with MDL updates.

11.3 Daily Performance Tests

11.3.1 Prior to initiating any data collection activities it is necessary to establish that a given GC/MS system meets the instrument performance criteria. This is accomplished through the analysis of 50 ng of p-bromofluorobenzene (BFB).

11.3.1.1 BFB must be analyzed at the start of every 12 hour sequence. 50 ng of BFB may be directly injected onto the GC column or purged in 5.0 ml of reagent water. BFB may not be analyzed simultaneously with a calibration standard.

11.3.1.2 The key ions produced during the analysis of BFB and their respective ion abundance criteria are given in Table 3.0. This criteria must be met before any calibration standards, blanks, or samples may be analyzed.

11.3.1.3 Use the target program to verify that the BFB spectrum is within criteria. If it is not within criteria, the analyst may use enhancing or other acceptable practices to put BFB within criteria.

11.3.1.4 If the criteria is not met, the BFB must be reanalyzed. Repeated failure shall require the instrument to be manually tuned. After manual tuning, the BFB must be re-injected and the abundance criteria must be met before proceeding.

11.3.2 After the instrument performance criteria is met, the initial calibration curve must be verified through the analysis of a continuing calibration at 50 ug/L or 20 ug/L. The continuing calibration criteria must be met before any method blank or sample analyses may proceed.

11.3.3 A method blank consisting of 5ml of reagent water spiked with 25 ug/L of IS and SMC's must be analyzed every 12 hours after calibration criteria has been met. An acceptable method blank must meet the following criteria:

PQL's can vary on each project. The PQL's must be reviewed prior to sample analysis It is essential that the analyst is aware of the specific requirements for each project.

Up to **three times** the PQL for method blanks for the target compounds Methylene chloride and Acetone.

- **Two times** the average MDL for all each of the other target compounds (average MDL currently is 0.5 for waters and 1.5 for soils)

Sample analysis may not proceed until the above method blank criteria has been met.

All volatile analyses associated with a method blank that does not meet the above requirements must be repurged, reanalyzed, and reported.

11.4 Matrix Spike, Matrix Spike Duplicates and Matrix Spike Blanks

11.4.1 An MS/MSD must be analyzed for each group of samples of a similar matrix within each case, 20 samples, group of samples of a similar concentration level (soils only), or each 7 calendar day period; whichever is more frequent. MSB's are required for NYSDEC protocols.

11.4.2 The limits for matrix spike compound recovery and relative percent difference (RPD) are given in Table 4.0. These limits are only advisory; therefore, no further action is required if the criteria limits are not achieved. However, frequent failures shall be investigated for possible laboratory generated error. Surrogate recovery can be outside of the laboratory generated windows in the MS/MSD/020ppb_QCS.

11.5 QC Check Samples are applicable to this method. This solution is run at a concentration of 20ppb for 8260B or 10ppb for 8260LL. This QCS is run after the daily method blank and before sample analysis. The 20ppb_QCS recoveries must fall within the laboratory generated guidelines. (see table 9.0 for water limits and 9.0a for soil limits) Up to four compounds can be outside of the recovery windows. If TCLP samples are being analyzed the analyst must use the TCLP blank fluid for the 20ppb spike. This will be called a 20ppb_LCS instead of 20ppb_QCS. If a reduced compound list is being analyzed, only the target compounds of concern must meet criteria. Various miscellaneous compounds are not included in the independent source QCS and therefore not controlled.

11.6 System Monitoring Compounds

11.6.1 SMC's are added to each sample, blank, standard, QCS and MS/MSD/MSB, prior to purging or extracting at 25 ug/L for waters, 25 ug/Kg for low level soils, and 2500 ug/Kg for medium level soils.

11.6.2 SMC recoveries must be within the QC limits given in Table 5.0. If the recovery for any one SMC is not within limits, the following are required:

- Check all calculations for accuracy, spiking solutions, and internal standards
- Reanalyze the sample if none of the above steps reveal a problem
- If an undiluted analysis with acceptable SMC recoveries is being submitted, do not reanalyze diluted samples if the SMC recoveries are outside the limits
- Never reanalyze the FMS, FMSD,or the 020ppb_QCS even if the SMC recoveries are outside the limits
- If the sample associated with the MS/MSD does not meet specifications, it should be reanalyzed only if the MS/MSD SMC recoveries are within the limits. Document in the narrative the similarity in the SMC recoveries between the sample and associated MS/MSD.

If the reanalysis of the sample solves the problem, then only submit the second analysis. If the reanalysis does not solve the problem, then submit the data from both analyses.

11.6.3 If the recovery of any one SMC in a method blank is outside limits, then the method blank and all associated samples must be reanalyzed.

11.7 Internal Standards

11.8.1 IS's are added to each sample, blank, standard, and MS/MSD/MSB, at 25 ug/L at the time of purging.

11.8.2 The retention times (RT) and extracted ion current profile (EICP) of each IS must be evaluated for all standards immediately after the data acquisition. The IS EICP areas must be monitored and evaluated for each sample, blank, MS, MSD. If the IS EICP changes by more than a factor of 2 (-50% to +100%) from the latest (12 hour) calibration standard, the GC/MS system must be inspected for malfunctions, and corrections made as required. If the RT for any IS changes by more than 30 seconds from the latest (12 hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. For samples analyzed within the same 12 hour time period as the initial calibration standards, compare the IS responses and RT's against the 50 ug/L calibration standard. When corrections are made, reanalysis of the samples analyzed while the system was malfunctioning is necessary.

If IS criteria is not within limits, the following are required:

- Check all calculations for accuracy, spiking solutions, and internal standards
- Reanalyze the sample if none of the above steps reveal a problem
- If the sample associated with the MS/MSD does not meet specifications, it should be reanalyzed only if the MS/MSD IS criteria is within the limits
- If the reanalysis of the sample solves the problem, then only submit the second analysis. If the reanalysis does not solve the problem, then submit the data from both analyses.

11.8 Quality Control Check Points

11.8.1 Analysis quality control approval report

Specific quality control check points have been established for the analysis of samples which are monitored through a Quality Control Approval Report (QCAR). The specific check points must be initialed and dated by the analyst to ensure the consistency and accuracy of the data produced. Refer to Figure 1.0 for the QCAR and specific control points covered.

11.8.2 Specific quality control check points have been established for the preparation of data deliverables which are monitored through a Quality Control Approval Report (QCAR). The specific check points must be initialed and dated by the analyst to ensure the consistency and accuracy of the data produced. Refer to Figure 1.1 for the QCAR and specific control points covered.

11.9 Analytical Documentation Procedures

11.9.1 Instrument batches

An instrument batch is created for each analytical sequence to organize all the associated data. Batch designations are of the format:

Xyynnnn.b

Where:

X = instrument identifier
Yy= last two digits of year

nnnn = file number of first calibration check standard in the batch for that day.

(i.e. T030012.b)

Instrument batches are number according to daily calibration check standard file. Therefore, the unique batch identifier can identify each analytical sequence. The batch consists of a file folder with all the associated QC information for the analytical sequence. The raw data is then bound together with the file folder to complete the batch. The batch, including the electronic logbook page will be scanned and electronically stored for future retrieval.

11.9.2 Filing system

All active batches are filed chronologically according to instrument. The batches are transferred to file boxes for long-term storage once all the associated data within a batch has been completed.

11.9.3 Data archiving

All data files, including the BFB analysis data file, are archived daily using the Target server back-up system. Care shall be exercised when purging data off the hard drives. To ensure that all data being purged has been archived, check latest archive date prior to purging any data.

11.9.4 Instrument run logs

It is TESTAMERICA's policy that all measurement data be recorded in logbooks with black ink, or electronic logbooks that are bound after analysis. Transcriptions shall be avoided whenever possible. The record shall reflect the measurement performed and all

appropriate details for conclusions related to the measurement. The record shall be signed and dated by the individual performing the measurement on the day the measurement is performed. Corrections shall be made by drawing a single line through the error, and initialing and dating the correction. A secondary authorization of the logbook is required and shall be performed by the department's manager or designee.

Currently in Volatiles, each instrument has its own three ring binder to maintain the electronic logbook pages until 90 pages are accumulated, then the logbook pages are given to QC to bind. All pages are sequentially numbered and paginated and secondary review done prior to daily scanning. Current run logs are held in the laboratory until they have been filled, for future reference. Older run logs shall be given to the QC officer for archiving. Each analytical sequence shall be started on a new page of the log and continued on the next page, if necessary. The header information designating the standard codes used shall be completed for each sequence. All standards used are recorded in this field for future traceability. The data file, job number, sample number, quantitation factor, dilution factor, analyst's signature, and date are recorded. Refer to Figure 2.0.

11.9.5 Initial data review sheet (short exception report on the target system)

The initial data review sheet (IDRS) is a computerized review sheet, which is used to check the key quality control criteria for compliance. The IDRS is used to check that all samples have been analyzed with the required calibration time frame. The IDRS is also used as the initial data review tool. Each sample is listed on the sheet and it is either accepted or rejected in the right hand column, by the analyst performing the data review. If reruns are required for dilutions, then the analyst shall indicate the proper dilution required for reanalysis. The data reviewer initial's and dates the IDRS. The batch is then filed when completed for use during deliverables preparation. Refer to Figure 3.0 for an example of the IDRS.

11.9.6 Electronic Corrective action reports need to be issued in a timely fashion. Non Conformance Module in the Lims system is used for quality control issues that need to be addressed in the Sample delivery group Narrative.

The Non - conformance report (NCM) is issued when a problem is encountered during analysis, data reduction or deliverables preparation, data validation, or when any deviations from this SOP occur. The NCM is prepared by the analyst first identifying the problem and is then electronically submitted to the department's manager for approval. The manager will review the NCM via e-mail.

Currently, for items beyond the realm of the NCM will require a corrective action report (CAR) this is for miscellaneous items the can not be covered by an NCM. The CAR is then redistributed to all the departments and individuals involved. Refer to Figure 4.0.

11.9.7 Chain of custody record

When samples are removed from storage for preparation or analysis they must be signed out utilizing the chain of custody record (COC). The samples shall then be signed back in on the COC upon their return to storage or designated "used" if the sample volume is consumed during the preparation or analysis.

11.9.8 Sample tracking record

Samples are tracked on the TestAmerica Lims Status reports. These reports are generated by the analyst as needed. Sample backlogs are generated often to ensure all samples are accounted for by each protocol. When samples are analyzed the analyst will update the Status report with the corresponding data files for each sample, and any relevant comments needed to assist reporting the data. NCM numbers will be recorded on the Status sheets to ensure all information is passed on to the client correctly. Samples requiring reanalysis are also documented as to the reanalysis dilution required, if any. Refer to Figure 5.0.

12.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

12.1 Instrumental Conditions

Instrument conditions will vary instrument to instrument, below is an example of the conditions. If the information is in bold print, then this condition will not vary due to method restrictions.

12.1.1 Purge & Trap Device

Purge Conditions:

	(VOACARB)
Purge Gas:	Helium
Purge Time:	**11.0 min**
Purge Flow:	**40 ml/min**
Dry Purge:	0.5 min
Purge Temp:	Ambient for LLW and MLS

Desorb Conditions:

	(VOACARB)
Desorb Temp:	250□C
Desorb Flow:	15 ml/min
Desorb Time:	0.5 min

Trap Reconditioning Conditions:

(VOACARB)

Reconditioning Temp:	270□C
Reconditioning Time:	8.0 min

12.1.2 Gas Chromatograph

Carrier Gas:	Helium
Flow Rate:	5 ml/min
Initial Temp.:	30□C
Initial Hold:	4 min
Ramp Rate 1:	5□C/min
Second Temp.:	100□C
Ramp Rate 2:	12□C/min
Final Temp.:	200□C
Final Hold:	1.0 min
Transfer Temp:	185□C

12.1.3 Mass Spectrometer

Electron Energy:	70 eV
Mass Range:	35 - 300 amu
Scan Time:	less than 1 sec/scan

The mass spectrometer must be tuned to meet the instrument performance check criteria for 50 ng of BFB listed in Table 3.0.

12.2 Sample Analysis Procedures

12.2.1 Water Samples (using guidance from method 5030B)

Samples are removed from Sample Control storage and are signed out in the chain of custody form. The chain of custody is a legal document, therefore there ensure all samples are signed out prior to leaving sample control. If a sample is brought to you directly from sample control ensure that the sample was signed out at that time.

All samples are allowed to warm to room temperature.

Make sure all instrumental operating conditions are correctly set and BFB, calibration and blank criteria have been met.

In a gas tight 5 ml syringe, load a 5 ml aliquot of sample (or 25 ml depending on request) by drawing up past the 5ml line on the syringe, then bring to volume. The sample that is expelled from the syringe to waste can be used to check and record the pH in the logbook. This procedure destroys the integrity of the sample for future analysis, therefore, if there is only one vial, the analyst shall fill a second gas tight syringe in the same manner. This second syringe is maintained only until such time as the analyst has determined that the first sample has been properly analyzed. If an analysis is required from the second syringe, it must be performed within 24 hours. Care must be taken to prevent air from leaking into the syringe during storage.

If the sample is being loaded on a Archon auto sampler, just remove excess label material from vial so the vial slips into the auto sampler position with ease, remove any label that may be on the cap to ensure the robotic arm won't stick to the label. Load vials and record client id's from actual vial into the logbook. Then program the Archon for correct method to run sequence. Internal standards and surrogates can be added automatically by Archon, or spiked at correct concentration into each vial. Record pH in logbook .

For 5ml purge using a syringe to load sample, spike the sample with 5 uL of the IS and SMC 25ug/ml spiking solution utilizing a 25 uL gas-tight, through the open syringe valve, then load the syringe contents into the sparging vessel. If an autosampler is utilized, set up the autosampler start and stop positions for the sequence being purged.

Inject the sample into the purging chamber and purge at ambient temperature. After purging, the sample is thermally desorbed onto the GC column.

While the trap is being desorbed into the GC, empty the purging chamber either manually or with the autodrain option. Wash the chamber with a minimum of two 5 ml flushes of reagent water to avoid carryover of target compounds.

The trap is then reconditioned while the sample is temperature programmed on the GC to separate the volatile organics.

When a sample has been analyzed that has saturated ions from a compound, sample analysis may not resume until a blank has been analyzed that demonstrates that the system is free of interferences. Once the system is free of interferences, the sample that saturated the detector must be diluted and reanalyzed.

If a sample is analyzed which contains target compounds at concentrations greater than the initial calibration upper limit, but not saturated, then the sample must be reanalyzed at an appropriate dilution. The purge and trap system shall be demonstrated to be free from carry-over through the subsequent analyses of blanks and/or samples which do not contain the target compound at a concentration greater than the PQL.

When the system is run unattended, using the autosampler, if a sample is analyzed which

contains target compounds at concentrations greater than the initial calibration upper limit, then the sample must be reanalyzed at an appropriate dilution. The samples analyzed subsequently shall be carefully evaluated. If any subsequent analyses contains the target compounds which were at concentrations greater than the initial calibration upper limit in the previous sample, at a concentrations greater than the PQL, then those samples must be reanalyzed once the system has been decontaminated and shown to free of interferences.

A water FMS/FMSD (FMSB-NYSDEC) is prepared by spiking the sample aliquot with 44 uL of calibration solution using a 50 uL gas-tight syringe. The spiked sample is then analyzed as previously described.

A method blank must be analyzed every 12 hours after the calibration criteria has been achieved. The method blank consists of 5 ml reagent water spiked with 5 uL of IS and SMC, and carried through the analytical procedure. Method blank criteria is defined in section 11.3.3.

12.2.2 Low Level Soil Samples (using guidance from method 5035)

The type of sample container that was used by the client must be determined at time of sample receipt. A hermetically sealed voa vial with preservative can be held for 14 days from collection. A sealed soil plug sample such as the EnCore sampler must be either transferred to a voa vial with 5mls of a Sodium Bisulfate solution, (**Note: using Sodium Bisulfate as a preservative has caused a "false" elevated level of acetone to be present due to a reaction between the soil and sodium Bisulfate. It is recommended to use reagent water if Acetone is a compound of concern on the site**) or reagent water and a magnetic stir bar, or analyzed within 48 hours from collection. If reagent water is used the vial must be frozen at a 45 degree angle to avoid cracking of the glass vial. This can be held for 14 days from collection. Ensure vial comes to room temperature prior to analysis. TESTAMERICA Connecticut prefers to use reagent water, since reactions can occur when adding soil sample to Sodium Bisulfide that cause moderate levels of Acetone to be falsely detected.

Samples are removed from Sample Control storage and are signed out on the chain of custody form. If the soil is received in a sealed plug type sampler such as the EnCore sampler, then the plug is transferred to a pre-weighed voa vial containing 5mls of the Sodium Bisulfate solution, or regent water, and a magnetic stir bar. The vial is sealed and not opened until time of disposal. The vial is then re-weighed and the weight is recorded on the vial along with the subtracted weight that determines the soil weight. The client ID is transferred along with the laboratory ID onto the voa vial. If QC has been requested on a particular sample the vials are labeled "MS" or "MSD". Once the soil is transferred and the above steps have been performed, the soil is placed in the volatile refrigerator for storage (freezer at 45 degree angle for reagent water vials).

Reagent water vial with stir bar will be used if the soil sample effervesces when it comes in contact with the Sodium Bisulfate solution. If the soil is place in water, this sample will either be run that day, or frozen at a 45 degree angle to prevent breakage, for up to 14 days.

The client / client service, must specify at time of bottle order if no preservative is required. Encore containers or equivalent may be used for low level soil analysis. If these are used the soil plug must be transferred within 48 hours from collection. **Arrangements must be made prior to shipping Encores to the laboratory for weekend transferring of the soil plugs.**

All samples are allowed to warm to room temperature. All soils must be re-weighed prior to analysis to determine weight of sample.

Make sure all instrumental operating conditions are correctly set and BFB, calibration and blank criteria have been met.

The sample consists of the entire contents of the sample container. The voa vial is not to be opened. This sample is then loaded on the Archon autosampler. The internal standards and surrogates along with 5mls of reagent water will be added automatically by the Archon, or by analyst, prior to the sample pre-heat mode. Then the contents of the voa vial will be purged onto the Voacarb 3000 trap, and analyzed by the Mass Spectrometer. Then the trap is reconditioned by heating.

Surrogates are spiked in all curve points, continuing calibrations, Laboratory control spikes (LCS's) method blanks and samples at 25ppb for low level soils.

A percent moisture determination is performed by weighing out 5 grams soil from a separate container that has been submitted for percent solid determination. The soil shall dry overnight at 105 degrees Celsius. Allow to the sample to cool before weighing the sample back. The percent moisture is determined according to the following equation:

$$\frac{\text{g of wet sample - g of dry sample}}{\text{g of wet sample}} \times 100$$

If a sample is analyzed which contains target compounds at concentrations greater than the initial calibration upper limit, then the high concentration method must be utilized, unless the client sent other sealed voa vials with a lower sample volume that would be within the linear calibration range. Then the sample must be reanalyzed at an appropriate dilution. No less than 1.0 gram of sample may be analyzed by the low level method (i.e. a 5 fold dilution) utilizing the low level soil method. If a larger dilution is required, then the medium level soil method must be employed. With Encore type samplers, dilutions can not easily be made. Most analyses that have compounds recovering over 200ppb will have

to be run as medium level soils, unless a portion of sample, no less than one gram was transferred from the Encore type sampler within the 48 hour holding time and analyzed as a low level soil.

The purge and trap system shall be demonstrated to be free from carryover through the subsequent analyses of blanks and/or samples which do not contain the target compound at a concentration greater than the PQL.(see section 9.2 for detail)

A low level soil FMS/FMSD (FMSB-NYSDEC) is prepared by spiking 50ppb of the calibration standard into two separate sample vials. The voa vials are not to be opened. This sample is then loaded on the Archon autosampler. The internal standards and surrogates along with 5mls of reagent water will be added automatically by the Archon prior to the sample pre-heat mode. Then the voa vial will be purged onto the Voacarb 3000 trap. Then analyzed by the Mass Spectrometer.

A low level soil method blank must be analyzed every 12 hours after the calibration criteria has been achieved. The method blank consists of 5.0 grams of a purified solid matrix added to 5.0 ml of reagent water and a magnetic stir bar. The method blank is then loaded on the Archon and automatically spiked with internal standards , surrogates ,and 5mls of reagent water then carried through the analytical procedure. Method blank criteria is defined in section 11.3.3.

12.2.3 Medium Level Soil Samples

Samples are removed from the Sample Control storage area and signed out on the chain of custody form.

All samples are allowed to warm to room temperature.

Make sure all instrumental operating conditions are correctly set and BFB, calibration and blank criteria have been met.

12.2.3.1 Laboratory Preserved

More than one scenario may take place with high concentration soil samples. Review the client's Quality Assurance Plan prior to preparing samples to ensure there are no other site requirements for preparing the methanol extracts. If the client expected high levels of volatiles in the field, then the samples were collected in the field preserved in methanol. The client may use the EnCore sampler or equivalent, therefore the plug of soil from the EnCore sampler is placed into the pre-weighed voa jar containing 10mls of purge and trap grade methanol. Record the weight of the jar, soil and methanol. Record the soil weight on the jar. Shake the jar for 2 minutes, then let the contents settle and transfer 1-2mls of the methanol extract into a extract vial. Store extract until time of analysis. It is also possible that a low level soil is actually a high level soil. Therefore the methanol

extraction is performed at the laboratory. If the sample is extracted at the laboratory, then extract as follows:

The sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. A medium level soil extract is prepared by weighing out 5.0 grams of sample into a 20 ml extraction vial, record the weight to the nearest 0.1 gram. Determine the percent moisture as in the low level method. Quickly add 10.0 ml of purge and trap grade methanol to the vial. Cap and shake for 2 minutes.

Using a disposable pipette, transfer about 1 ml of sample extract to a GC vial for storage. The remainder of the extract may be discarded. Transfer about 1 ml of the reagent methanol to a GC vial for use as the method blank. These extracts may be stored in the dark at 4 degrees Celsius (+/- 2 degrees) prior to analysis.

In a gas tight 5 ml syringe, load a 5 ml aliquot of reagent water and spike with 5 uL of IS/Surrogate spiking solution (25ppb on column concentration) using a 25 uL gas-tight syringe, and 100 ul of sample using a 250 uL gas tight syringe, extract and purge for 11.0 minutes at ambient temperature. If an autosampler is utilized, set up the autosampler start and stop positions for the sequence being purged. After purging, desorb onto the GC column.

The medium level soil extract is analyzed under a water initial and continuing calibration.(using guidance form method 5030B)

The trap is then reconditioned at 270 degrees Celsius for 6-8 minutes while the sample is temperature programmed on the GC to separate the volatile organics.

If an extract is analyzed which contains target compounds at concentrations greater than the initial calibration upper limit, then the extract must be reanalyzed at an appropriate dilution. Volumes of less than 10 ul (i.e. 10 fold MLS dilution) shall be prepared diluting an aliquot of the methanol extract and then taking 100 uL for analysis. Add the volume of methanol extract from the sample and a volume of clean methanol to total 100 ul. The total methanol volume added shall be 100 ul, excluding the methanol in the standards.

The purge and trap system shall be demonstrated to be free from carry-over through the subsequent analyses of blanks and/or samples which do not contain the target compound at a concentration greater than the PQL.

A medium level soil FMS/FMSD is prepared by spiking a 5.0 gram sample with 10.0 mls of purge and trap methanol. 880ul of the sample is added to the 44ml voa vial and then spiked with 44ul of calibration check standard. The spiked sample extract is then analyzed as previously described.

A method blank must be analyzed every 12 hours after the calibration criteria has been achieved. The method blank consists of 10.0 ml of reagent methanol. A 100 uL aliquot of the method blank extract is then spiked into 5 ml reagent water fortified with 5 uL IS/Surrogate solution. The method blank is then carried through the analytical procedure as described previously. Method blank criteria is defined in section 11.3.3.

12.2.3.2 Field Preserved

If high volatile concentrations are expected in the soil samples a different sampling method is required. The soil vial will contain methanol, be pre-weighed and labeled, prior to shipping to the client (Some states require surrogates to be pre-spiked in the methanol prior to sample collection. This can be done when requested. The laboratory must be notified for special requests) There must also be an empty soil vial for total solids determination in the laboratory. Five grams of soil will be added to the methanol vial in the field and weights recorded on the chain of custody, vials or other appropriate locations.

Oily waste samples will be collected in empty vials (unless known to be soluble in methanol or polyethylene glycol (PEG) * PEG must be requested when placing bottle orders otherwise vials will contain methanol. The laboratory will weigh the voa vial prior to analysis, record the weight in the logbook, check to ensure the methanol and soil mixture is still present. If not a corrective action must be written to notify client of possible re-sampling, or another acceptable solution. The sample is then shaken and allowed to settle. A portion of the diluted sample is then removed from the vial and analyzed by method 5030B.(see section 11.2.1) Total solids, sample weight, and sample volume used, all need to be recorded to determine correct dilution factor for quantitation.

The default volume of methanol for TESTAMERICA Connecticut is 10ml without surrogates if volumes are not specified by client

12.3 Qualitative Analysis

12.3.1 Target Compounds

The relative retention time of a target compound must be within +/- 0.06 RRT units of the RRT of the calibration standard for a positive identification. For reference the standard must be analyzed within the same 12 hour time period as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT shall be assigned by using the extracted ion current profiles for ions unique to the component of interest.

In addition, a comparison must be made between the mass spectrum obtained in the sample analysis and the reference mass spectrum for that compound, which was obtained on that specific GC/MS system. The requirements for qualitative verification by

comparison of mass spectra are as follows:

All ions present in the reference spectrum at an intensity greater than 10% must be present in the sample spectrum.

The relative intensities of the ions above 10% must agree with 20% between the reference and sample spectra.

Ions greater than 10% in the sample spectrum but not present in the reference spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by the above criteria, but in the technical judgement of the analyst, the identification is correct, then the compound shall be reported.

12.3.2 Tentatively Identified Compounds

If requested a library search shall be performed for non-target compounds in the sample for purposes of tentative identification. For this purpose, the most recent release of the NIST mass spectral library shall be used.

Up to 10 organic compounds of apparent concentration not listed in Table 1.0, shall be tentatively identified via a forward library search. Only compounds with responses greater than 10% of the closest IS exhibiting no interference are to be searched.

The Target software is utilized to perform the automated library search. The program (TICS) is executed with the data file, method, and number of compounds to be searched, specified for each sample or blank. Prior to running the program, the analyst must delete from the quantitation file, using the Target review program, the non-TCL compounds which were identified in the quantitation file. This will facilitate their automated search using the program. If the non-TCL positive hits are not removed prior to executing the program, they would be counted as target compounds and not be searched by the program, leading to false negatives.

A tentative identification will be made after a comparison between the mass spectrum obtained in the sample analysis and the library search mass spectra found for that compound. The requirements for tentative verification by comparison of mass spectra are as follows:

Ions present in the reference spectrum at an intensity greater than 10% should be present in the sample spectrum.

The relative intensities of the ions above 10% should agree with 20% between the reference and sample spectra.

Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or coeluting compounds.

Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible background subtraction by the data system.

If in the technical judgement of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound shall be reported as unknown. Additional classification shall be made if possible (i.e. Unknown hydrocarbon).

12.4 Quantitative Analysis

12.4.1 Target Compounds

Target compounds are quantitated by the internal standard technique. The associated internal standard used is listed in Table 6.0. The EICP area of the quantitation ions of compounds listed in Tables 8.0 are used. The quantitation ion for the SMC compound bromofluorobenzene can be m/z 174 instead of m/z 95 due the co-elution of the target compound 1,1,2,2-tetrachloroethane, which interferes with m/z 95. Other co-eluters that must be inspected are listed in Figure 7.0.

The relative response factor (RRF) from the initial calibration standard or the equation for linear regression is used to calculate the concentration in the sample depending on the percent RSD in the calibration curve. When compound concentrations are below the PQL, but the compound meets identification criteria, report the concentration with a "J" qualifier.

Water Samples

Concentration

$$ug/L = \frac{(Ax)\,(Is)\,(Df)}{(Ais)\,(RRF)\,(Vo)}$$

where,

Ax = area of the compound quantitation ion

Ais = area of IS quantitation ion

Is = IS amount in nanograms

RRF = Average Relative response factor from the ambient temperature purge of the Initial calibration curve (Linear Regression may replace this equation if the mean

response for all the compounds in the method is above 15% RSD in initial calibration)

Vo = volume of water purged in ml's

Df = Dilution factor. The dilution factor for analysis of water samples for volatiles by this method is defined as the ration of the number of milliliters (ml) of water purged (i.e. Vo above) to the number of ml of the original water sample used for purging. For example, if 2.5 ml of sample is diluted to 5.0 ml with reagent water and purged, DF=5.0 ml/2.5 ml = 2.0. If no dilution is performed, Df = 1.0.

Low Level Soil Samples

Concentration
(dry weight basis)

$$ug/Kg = \frac{(Ax)(Is)}{(Ais)(RRF)(Ws)(D)}$$

where,

Ax, Is, and Ais are as given for water.

$$D = \frac{100 - \%\ moisture}{100}$$

Ws = weight of sample added in grams

RRF = Average Relative response factor from the heated temperature purge of the Initial calibration curve. (Linear Regression may replace this equation if the mean response for all compounds in the method is above 15% RSD in initial calibration)

Medium Level Soil Samples

Concentration
(dry weight basis)

$$ug/Kg = \frac{(Ax)(Is)(Vt)(1000)(Df)}{(Ais)(RRF)(Va)(Ws)(D)}$$

where,

Ax, Is, Ais and RRF are as given for water.

$$D = \frac{100 - \%\ \text{moisture}}{100}$$

RRF = Average Relative response factor from the ambient temperature purge of the initial calibration curve. (Linear Regression may replace this equation if the mean response for all compounds in the method is above 15% RSD in initial calibration)

Ws = weight of sample extracted in grams

Vt = total volume of methanol extract in ml

Va = volume of the methanol extract added to the reagent water for purging in ul

Df = Dilution factor. The dilution factor for medium level soils is defined as the ratio of the number of microliters (uL) of methanol added to the reagent water for purging (i.e. Va above) to the number of uL of the methanol extract of the sample contained in that volume Va. The dilution factor is equal to one in all cases other than those requiring dilution of the methanol extract.

12.4.2 Linear Fit

$$Conc = A + B\frac{(R_x C_{is})}{R_{is}}$$

Conc = Concentration in ug/l
R_x = Response for analyte(area of quantitation ion)
R_{is} = Response for internal standard(area of quantitation ion)
C_{is} = Concentration of internal standard
A = Intercept
B = Slope

The corresponding Target software calculation is as follows:

$$Conc = C_{is}\left(b + \frac{1}{m1} \times \frac{R_x}{R_{is}}\right)$$

b = Concentration Ratio Intercept
m1 = Inverse of slope

12.4.3 Quadratic fit

$$\text{Conc} = A + B\,\frac{(R_x C_{is})}{R_{is}} + C\,\frac{(R_x C_{is})^2}{R_{is}}$$

C = Curvature

The corresponding Target software calculation is as follows:

$$\text{Conc} = C_{is}\left(b + m1 \times \frac{R_x}{R_{is}} + m2 \times \left(\frac{R_x}{R_{is}}\right)^2\right)$$

m1 = First order coefficient
m2 = Curvature(Second order coefficient)

12.4.4 The concentration in the sample is then calculated.

12.4.5 Tentatively Identified Compounds

An estimated concentration for non-target compounds tentatively identified in the sample shall be determined by the internal standard method. For quantitation, the nearest IS free of interferences shall be used.

The equation for calculating concentrations are the same as in 12.4.1. Total area counts from the total ion chromatograms are used for both the IS and compound. A RRF of 1.0 is assumed and the resulting concentration shall be qualified as "J" (estimated), indicating the quantitative and qualitative uncertainties associated with this non-target compound.

12.4.6 The three Xylene isomers are to be reported as Xylenes (total). The meta and para isomers coelute on the capillary column, therefore, special attention must be given to their quantitation. The two reponse factors (RF) for the three Xylene isomers are totaled divided by two and an average RF is used for quantitation.

12.4.7 The cis and trans isomers of 1,2-Dichloroethene are to be reported as 1,2-Dichloroethene (total). The two isomers do not coelute on the capillary, therefore, the RRF is determined by summing the two isomer areas and then dividing by the total isomer concentrations. The area from both peaks and this RRF are then used to quantitate the 1,2-Dichloroethene (total) concentration. Both isomers must be present in the initial and continuing calibration standards.

12.4.8 If the on-column concentration of any compound in any sample exceeds the initial calibration range, a new aliquot of that sample must be diluted and purged. Guidance in performing dilutions, and exceptions to this requirement are as follows:

12.4.9 Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range.

12.4.10 The dilution factor chosen shall keep the response of the largest analyte peak for a target compound in the upper half of the initial calibration range of the instrument.

12.4.11 Data for more than two analyses shall not be submitted.

12.4.12 Run associated sample at correct dilution prior to running FMS/FMSD. It is not necessary to re-run a FMS/FMSD, or 020ppb_QCS if internal standards and or surrogates are out of criteria. Or spike compounds are outside of recovery windows. However; FMSB's Must meet Internal standard and surrogate criteria. Since a Full Matrix spike is used, the likely hood of all compounds being within the acceptable range are slim. Therefore, note the compounds outside of the laboratory windows in the SDG narrative.

12.5 Instrument Maintenance

12.5.1 Preventative maintenance

All HP instrumentation is covered by a service contract with an external instrumentation service vendor, or by TESTAMERICA personnel trained in preventative maintenance. Preventative maintenance is perform at scheduled intervals on all equipment according to the frequency detailed in Appendix A. All instrument preventative maintenance is performed according the manufacturers recommended procedures, by trained personnel. All preventative maintenance shall be thoroughly documented in the maintenance log (see Figure 6.0), as to a description of the maintenance performed, the date performed, and the personnel performing the maintenance. Upon returning the system into control, a cross-reference batch number shall be recorded in the maintenance log. This batch number will demonstrate that a successful repair has been achieved.

12.5.2 Corrective maintenance determinants and procedures

Corrective maintenance is deemed necessary when the analytical system, after reanalysis, cannot meet tune, calibration, or other protocol specific QC criteria. Corrective maintenance may include, but is not limited to, decontamination of the system, injection port cutting and cleaning, source cleaning, replacing the electron multiplier, column replacement, jet separator cleaning or replacement, or filament replacement. All corrective maintenance is performed according the manufacturers recommended procedures, by trained personnel. All corrective maintenance shall be thoroughly documented in the maintenance log, as to a description of the maintenance performed, the date performed, and the personnel performing the maintenance.

12.5.3 Maintenance authorization

All preventative and corrective maintenance is authorized by the department's manager, or designee. When outside maintenance is deemed necessary, a service call is placed for all equipment covered under a service contract, by the department's manager, or designee.

12.6 Data System

12.6.1 Data Acquisition and System Operation

Data is acquired from sample analyses using the Target software. Analytical batches are set up with all the associated sample ID, dilution, and data file information. Automated post-acquisition quantitation is qued with the appropriate method, as well as, post-acquisition archiving of the data file. The sequence is assigned and started using the ChemStation software.

12.6.2 Instrument errors

System errors are logged to the ChemStation error logs. The system manager shall be responsible for checking and providing corrective actions for all major system errors. Minor system errors, such as insufficient disk space, are handled by trained analysts, as necessary.

12.6.3 Manual Integrations and Editing Flags
Manual integrations are required when the automated software doesn't correctly integrate extracted ion current profiles (EICP). Manual Integrations are performed. The target compound number of interest is selected and the EICPs are graphically presented. The peak can then be correctly integrated. A new quantitation report is produced. The manually integrated data file is the saved by exiting and saving from file edit. Manual integrations are flagged by the data system with the "M" qualifier. Troublesome integrations are on file for each instrument that are routinely seen due to column conditions and interfering secondary ions from other compounds.

13.0 CALCULATIONS

13.1 Relative Response Factor (RRF)

$$RRF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

where,

Ax = area of the compound quantitation ion

Ais = area of IS quantitation ion

Cis = IS concentration

Cx = compound concentration

An average RRF is calculated for each compound and SMC from the initial calibration.

The RRF used for quantitation is based upon the ortho-Xylene isomer peak. The area from both peaks and this RRF are then used to quantitate the Xylenes (total) concentration. All three isomers must be present in the initial and continuing calibration standards.

The RRF is used for 1,2-Dichloroethene is calculated by summing the two isomer areas and then dividing by the total isomer concentrations. The area from both peaks and this RRF are then used to quantitate the 1,2-Dichloroethene (total) concentration in a sample. Both isomers must be present in the initial and continuing calibration standards.

13.2 Percent Relative Standard Deviation (%RSD)

$$\%RSD = \frac{\text{standard deviation}}{\text{mean}} \times 100$$

13.3 Percent Difference (%D)

$$\%D = \frac{(\text{average RRFi}) - (\text{RRFc})}{(\text{average RRFi})} \times 100$$

where,

average RRFi = average RRF from the initial calibration

RRFc = RRF from the continuing calibration standard

13.4 Percent Moisture

$$\%\ \text{moisture} = \frac{\text{g of wet sample} - \text{g of dry sample}}{\text{g of wet sample}} \times 100$$

13.5 Target Compound Concentrations

The calculations used to determine the target compound concentrations are described in section 11.4.

13.6 SMC Percent Recovery

$$\% \text{ Recovery} = \frac{\text{concentration found}}{\text{concentration spiked}} \times 100$$

13.7 Matrix Spike Recovery

$$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

where,

SSR = spiked sample result

SR = sample result

SA = spike added

13.8 Relative Percent Difference

$$RPD = \frac{\text{absolute } (MSR - MSDR)}{(½)(MSR+MSDR)} \times 100$$

where,
MSR = matrix spike recovery

MSDR = matrix spike duplicate recovery

The absolute value of the recovery difference is used in the above equation.

13.9 Adjusted Contract Required Quantitation Limit for Samples

$$\text{Adjusted PQL} = \frac{(PQL) \times Df}{D}$$

where,

$$D = \frac{100 - \% \text{ moisture}}{100}$$

Df = the dilution factor

14.0 ACCEPTANCE OF DATA

14.1 Method Blank

The method blank can contain analytes up to the PQL for the target compounds, except

methylene chloride and acetone which must be less than or equal to three times the PQL. Special projects may require lower detection limits than the PQL. In these cases the method blank must not contain compounds over the client requested detection limit.(See Table 10.0 for various method blank criteria for special projects.)

If a method blank exceeds the limits for contamination above, the laboratory shall consider the analytical system to be out of control. The source of the contamination must be investigated and appropriate corrective actions taken and documented before further sample analysis proceeds.

14.2 System Monitoring Compounds (SMC)

All SMC's must be within the recovery criteria listed in Table 5.0. Method blanks and samples with recoveries outside the required windows must be reanalyzed. Refer to section 11.6 for SMC information.

14.3 Instrument Performance Check

The criteria for bromofluorobenzene is listed in Table 3.0 and in section 11.3.1.

14.4 Internal Standards
The IS criteria is described in section 11.7.

14.5 Full Matrix Spike/Full Matrix Spike Duplicate

The FMS/FMSD criteria is described in section 11.4.

15.0 REPORTING OF RESULTS

Refer to documentation policy/procedures

16.0 Pollution Prevention

16.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

16.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

16.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and

degradation.

16.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

16.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

16.1.5 Chemical /material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

17.0 Waste Management

17.1 All waste shall be managed in accordance with all state and federal requirements, TESTAMERICA Connecticut's Hazardous waste management plan, the RCRA Contingency Plan and the Corporate Safety Manual.

17.2 All personnel who handle or generate waste must be trained prior to handling waste.

- Aqueous waste water is put in screw cap 500ml bottles with sodium bisulfate to neutralize water. This water can be put in 50 gallon drum for neutralizing and disposal as treated waste.
- Soil waste is initially stored in satellite waste area's below desks in the volatiles laboratory. This soil is then put into the non hazardous soil waste drums in the hazardous waste room.
- Methanol waste from medium level soils samples are initially accumulated and segregated from low level soil waste in the satellite waste area below the desks in the volatiles laboratory. This soil / methanol waste and vial is disposed of in flammable waste vials drum in the hazardous waste room.
- Outdated, expired and obsolete calibration standards are laboratory packed yearly.

18.0 SUPPLEMENTAL DOCUMENTS

18.1 Standard Operating Procedure Documentation Policy/Procedures Organics, 18.2 SOP for Volatile Standards Preparation

18.3 SOP for Samples Processing Methods Performed at Sample Arrival

18.4 SOP for Log-In Methods for CLP Samples

18.5 SOP for Storing Water and Soil Samples for Organic and Inorganic Sample Analysis

18.6 SOP for Documenting Sample Removal from the Laboratory

18.7 Standard Operating Procedure for Sample Tracking

19.0 REFERENCES

19.1 Purge & Trap Method EPA SW846 3rd Edition, Methods 5030B/5035.

19.2 Volatile organics by Gas Chromatography/Mass Spectrometry (GC/MS) SW846 3rd Edition, Method 8260B.

19.3 EnChem, Inc 1795 Industrial Drive, Green Bay WI 54302 (920) 469-2436
EnChem produces the EnCore sampler a tool that one manually inserts into the soil to take a plug of soil sample. The sample is then capped and shipped to the laboratory. The laboratory then must remove the soil and analyze by either the 5035 soil method or a methanol extraction method (5030B) * soil samples must be analyzed within 48 hours after collection, or transferred to vials. (section 6.2.1.8 5035-10)

19.3 Associated Design and Manufacturing Company, 814 North Henry Street, Alexandria, VA 22314 (703) 549-5999 Purge and Trap Soil Sampler - (model 3780PT) this tool allows a sampler to take a soil sample plug and through the use of various adapters for the voa vial, put the plug of soil into a standard 40ml voa vial, then ship the voa vial and voa vial adapter to the laboratory for analysis by method 5035.

19.4 Becton Dickinson & Co Franklin Lakes NJ 07417-1884 (888) 237-2762 or VWR, 800) 932-5000 Sterile syringe (not cut). The end of the plastic syringe barrel is cut off, either in the field, or by the laboratory prior to shipping. The syringe is tarred out in the field on a portable balance prior to scooping up a sample. Five grams of soil sample is then collected in the syringe and put into a voa vial that has been pre-weighed, preserved with sodium bisulfate and contains a Teflon coated stir bar(all supplied by laboratory). Vials are sealed and samples are directly loaded onto an autosampler which pierces the septa of the voa vial therefore keeping the sample sealed and not opened by the laboratory. Four other vial should be collected in the same manner to ensure enough sample volume for repeated analyses at the laboratory. One empty voa vial should also be filled with soil for total solids determination, and to be used if the soil collected must be run as a medium level soil.

20.0 SUBSTANTIVE REVISIONS

20.1 Original issue; lab update to method 8260B - January 27, 1998.

20.2 Revision on: November 17, 1998. Changed Laboratory name in entire document from AEN to STL. Section 7.0; updated instrumentation and computer systems. Section 11.0; added method 5035 low level soil procedure.

20.3 Corrected section 9.4.1 item number 2 to state "below 15%". Corrected header problem.

- January 27, 1999.

20.4 Updated to reflect A.C.O.E. modifications March 19,1999
20.5 Updated to reflect NELAP added sections 3,16,17 renumbered SOP October 6, 1999
20.6 Updated after Laboratory Manager's review 11/05/1999
20.7 Annual review of S.O.P. updated as per requirements of STL QC program 02/26/01.
20.8 Review of S.O.P. updated as per requirements of STL QC program 06/12/2003.
20.9 Updated to reflect changes in method 5035. 9/23/2003.
20.10 Updated to reflect low level water changes for 5973, and ACOE requirements 2/11/2005
20.11 General updates 2/16/2005
20.12 General updates 4/10/2007
20.13 General corrections found during group review of 8260B sop and updates to include TestAmerica name. 8/23/2007.

TABLES

TABLE 1.0
TARGET COMPOUND LIST (TCL) AND ESTIMATED QUANTITATION LIMITS (EQL)

Volatile Organics	Quantitation Limits*			
	Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	On Column ng
Chloromethane	10	10	1,000	(50)
Bromoethane	10	10	1,000	(50)
Vinyl Chloride	10	10	1,000	(50)
Chloroethane	10	10	1,000	(50)
Methylene Chloride	10	10	1,000	(50)
Acetone	10	10	1,000	(50)
Carbon Disulfide	5	5	500	(50)
1,1-Dichloroethene	5	5	500	(50)
1,1-Dichloroethane	5	5	500	(50)
1,2-Dichloroethene (total)	5	5	500	(50)
Chloroform	5	5	500	(50)
1,2-Dichloroethane	5	5	500	(50)
2-Butanone	10	10	1,000	(50)
1,1,1-Trichloroethane	5	5	500	(50)
Carbon Tetrachloride	5	5	500	(50)
Bromodichloromethane	5	5	500	(50)
1,2-Dichloropropene	5	5	500	(50)
cis-1,3-Dichloropropene	5	5	500	(50)
Trichloroethene	5	5	500	(50)
Dibromochloromethane	5	5	500	(50)
1,1,2-Trichloroethane	5	5	500	(50)
Benzene	5	5	500	(50)
trans-1,3-Dichloropropene	5	5	500	(50)
Bromoform	5	5	500	(50)
4-Methyl-2-pentanone	10	10	1,000	(50)
2-Hexanone	10	10	1,000	(50)
Tetrachloroethene	5	5	500	(50)
Toluene	5	5	500	(50)
1,1,2,2-Tetrachloroethane	5	5	500	(50)
Chlorobenzene	5	5	500	(50)
Ethylbenzene	5	5	500	(50)
Styrene	5	5	500	(50
Xylene (total)	5	5	500	(50)

***Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.**

TABLE 1.1
8260B COMPOUND LIST AND ESTIMATED QUANTITATION LIMITS (EQL)

Volatile Organics	Quantitation Limits*			
	Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	25 mL Purge ug/L
Benzene	10	10	1,000	(50)
Bromobenzene	10	10	1,000	(50)
Bromochloromethane	10	10	1,000	(50)
Bromodichloromethane	10	10	1,000	(50)
Bromoform	10	10	1,000	(50)
Bromomethane	10	10	1,000	(50)
n-Butylbenzene	5	5	500	(50)
sec-Butylbenzene	5	5	500	(50)
tert-Butylbenzene	5	5	500	(50)
Carbon Tetrachloride	5	5	500	(50)
Chlorobenzene	5	5	500	(50)
Chlorodibromomethane	5	5	500	(50)
Chloroethane	10	10	1,000	(50)
Chloroform	5	5	500	(50)
Chloromethane	5	5	500	(50)
2-Chlorotoluene	5	5	500	(50)
4-Chlorotoluene	5	5	500	(50)
1,2-Dibromo-3-chloropropane	5	5	500	(50)
1,2-Dibromoethane	5	5	500	(50)
Dibromomethane	5	5	500	(50)
1,2-Dichlorobenzene	5	5	500	(50)
1,3-Dichlorobenzene	5	5	500	(50)
1,4-Dichlorobenzene	5	5	500	(50)
Dichlorodifluoromethane	5	5	500	(50)
1,1-Dichloroethane	10	10	1,000	(50)
1,2-Dichloroethane	10	10	1,000	(50)
1,1-Dichloroethene	5	5	500	(50)
cis-1,2-Dichloroethene	5	5	500	(50)
trans-1,2-Dichloroethene	5	5	500	(50)
1,2-Dichloropropane	5	5	500	(50)
1,3-Dichloropropane	5	5	500	(50)
2,2-Dichloropropane	5	5	500	(50
1,1-Dichloropropene	5	5	500	(50)

*Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

TABLE 1.1 (continued)
8260 COMPOUND LIST AND ESTIMATED QUANTITATION LIMITS (EQL)

Volatile Organics	Quantitation Limits*			
	Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	25 mL Purge ug/L
Ethylbenzene	10	10	1,000	(50)
Hexachlorobutiene	10	10	1,000	(50)
Isopropylbenzene	10	10	1,000	(50)
p-Isopropyltoluene	10	10	1,000	(50)
Methylene Chloride	10	10	1,000	(50)
Naphthalene	10	10	1,000	(50)
n-Propylbenzene	5	5	500	(50)
Styrene	5	5	500	(50)
1,1,1,2-Tetrachloroethane	5	5	500	(50)
1,1,2,2-Tetrachloroethane	5	5	500	(50)
Tetrachloroethene	5	5	500	(50)
Toluene	5	5	500	(50)
1,2,3-Trichlorobenzene	10	10	1,000	(50)
1,2,4-Trichlorobenzene	5	5	500	(50)
1,1,1-Trichloroethane	5	5	500	(50)
1,1,2-Trichloroethane	5	5	500	(50)
Trichloroethene	5	5	500	(50)
Trichlorofluoromethane	5	5	500	(50)
1,2,3-Trichloropropane	5	5	500	(50)
1,2,4-Trimethylbenzene	5	5	500	(50)
1,3,5-Trimethylbenzene	5	5	500	(50)
Vinyl Chloride	5	5	500	(50)
o-Xylene	5	5	500	(50)
m-Xylene	5	5	500	(50)
p-Xylene	10	10	1,000	(50)

***Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.**

TABLE 2.0
APPENDIX IX COMPOUND LIST AND ESTIMATED QUANTITATION LIMITS (EQL)

Volatile Organics	Quantitation Limits* **		
	Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg
Chloromethane	10	10	1,000
Bromomethane	10	10	1,000
Vinyl Chloride	10	10	1,000
Chloroethane	10	10	1,000
Methylene Chloride	5	5	500
Acetone	10	10	1,000
Carbon Disulfide	5	5	500
1,1-Dichloroethene	5	5	500
1,1-Dichloroethane	5	5	500
1,2-Dichloroethene (total)	5	5	500
Chloroform	5	5	500
1,2-Dichloroethane	5	5	500
2-Butanone	10	10	1,000
1,1,1-Trichloroethane	5	5	500
Carbon Tetrachloride	5	5	500
Vinyl Acetate	10	10	1,000
Bromodichloromethane	5	5	500
1,2-Dichloropropane	5	5	500
cis-1,3-Dichloropropene	5	5	500
Trichloroethene	5	5	500
Dibromochloromethane	5	5	500
1,1,2-Trichloroethane	5	5	500
Benzene	5	5	500
trans-1,3-Dichloropropene	5	5	500
Bromoform	5	5	500
4-Methyl-2-Pentanone	10	10	1,000
2-Hexanone	10	10	1,000
Tetrachloroethene	5	5	500
1,1,2,2-Tetrachloroethane	5	5	500
Toluene	5	5	500
Chlorobenzene	5	5	500
Ethylbenzene	5	5	500
Styrene	5	5	500

***Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.**

TABLE 2.0 (continued)
APPENDIX IX COMPOUND LIST AND ESTIMATED QUANTITATION LIMITS (EQL)

Volatile Organics	Quantitation Limits * **		
	Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg
Xylene (total)	5	5	500
Dibromomethane	10	10	1,000
1,2-Dibromoethane (EDB)	10	10	1,000
1,1,1,2-Tetrachloroethane	10	10	1,000
1,2,3-Trichloropropane	10	10	1,000
Dichlorodifluoromethane	10	10	1,000
Iodomethane	10	10	1,000
3-Chloro-1-Propene	10	10	1,000
2-Methyl-2-Propenenitrile	10	10	1,000
2-Chloro-1,3-Butadiene	10	10	1,000
Methyl Methacrylate	10	10	1,000
Ethyl Methacrylate	10	10	1,000
1,4-Dichloro-2-Butene	10	10	1,000
1,2-Dibromo-3-Chloropropane	10	10	1,000
Acrolein	100	100	10,000
Acrylonitrile	35	35	3,500
Trichlorofluoromethane	10	10	1,000
Pentachloroethane	5	5	500

***Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.**

**** Chart has estimated PQLs. PQL numbers change with updates to MDL levels. (PQL is determined by three to five times current laboratory Method Detection limits MDL)**

TABLE 3.0
GC/MS PERFORMANCE STANDARD
BROMOFLUOROBENZENE (BFB)

m/z	Ion Abundance Criteria	% Relative Base Peak	Abundance Appropriate Peak
50	15-40% of mass 95	25.60	25.60
75	30-60% of mass 95	54.84	54.84
95	Base peak, 100% relative abundance	100.00	100.00
96	5-9% of mass 95	7.58	7.58
173	Less than 2 percent of mass 174	0.00	0.00
174	Greater than 50% of mass 95	90.01	90.01
175	5-9% of mass 174	6.66	7.40
176	95-101% of mass 1 74	88.81	98.66
177	5-9% of mass 175	6.52	7.32

TABLE 4.0
MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

Compound	% Recovery Water	RPD Water	% Recovery Soil	RPD Soil
1,1-Dichloroethane	61-145	14	59-172	22
Trichloroethene	71-120	14	62-137	24
Benzene	76-127	11	66-142	21
Toluene	76-125	13	59-139	21
Chlorobenzene	75-130	13	60-133	21

TABLE 5.0
SYSTEM MONITORING COMPOUND RECOVERY LIMITS*

Compound	% Recovery Water	% Recovery Soil
Toluene-d_8	85-125	54-93
Dibromofluoromethane	87-128	53-114
Bromofluorobenzene	83-120	50-103
1,2-Dichloroethane-d_4	78-118	50-121

*Surrogate windows will change as periodic updates of surrogate windows are performed.

TABLE 6.0
VOLATILE INTERNAL STANDARDS WITH CORRESPONDING TARGET COMPOUNDS
AND SYSTEM MONITORING COMPOUNDS ASSIGNED FOR QUANTITATION

Bromochloromethane	1,4-Difluorobenzene	Chlorobenzene-d_5
Chloromethane	1,1,1-Trichloroethane	2-Hexanone
Bromomethane	Carbon Tetrachloride	4-Methyl-2-Pentanone
Vinyl Chloride	Bromodichloromethane	Tetrachloroethene
Chloroethane	1,2-Dichloropropane	1,1,2,2-Tetrachloroethane
Methylene Chloride	trans-1,3-Dichloropropene	Toluene
Acetone	Trichloroethene	Chlorobenzene
Carbon Disulfide	Dibromochloromethane	Ethylbenzene
1,1-Dichloroethene	1,1,2-Trichloroethane	Styrene
1,1-Dichloroethane	Benzene	Xylene (total)
1,2-Dichloroethene (total)	cis-1,3-Dichloropropene	Bromofluorobenzene (smc)
Chloroform	Bromoform	Toluene-d_8 (smc)
1,2-Dichloroethane		
2-Butanone		
1,2-Dichloroethane-d_4 (smc)		

(smc) - system monitoring compound

TABLE 7.0
CHARACTERISTIC IONS FOR SYSTEM MONITORING COMPOUNDS AND INTERNAL STANDARDS FOR VOLATILE ORGANIC COMPOUNDS

System Monitoring Compounds	Primary Ion	Secondary Ion(s)
Dibromofluoromethane	111	113,192
4-Bromofluorobenzene	95	174,176
1,2-Dichloroethane-d_4	65	102
Toluene-d_8	98	70, 100

Internal Standards	Primary Ion	Secondary Ion(s)
Fluorobenzene	96	50,70
1,4-Dichlorobenzene d4	152	63, 88
Chlorobenzene-d_5	117	82, 119

TABLE 8.0
CHARACTERISTIC IONS FOR VOLATILE TARGET COMPOUNDS

Analyte	Primary Ion*	Secondary Ion(s)
Chloromethane	50	52
Bromomethane	94	96
Vinyl Chloride	62	64
Chloroethane	64	66
Methylene Chloride	84	49, 51, 86
Acetone	43	58
Carbon Disulfide	76	78
1,1-DIchloroethene	96	61, 98
1,1-Dichloroethane	63	65, 83, 85, 98, 100
1,2-Dichloroethene	96	61, 98
Chloroform	83	85
1,2-Dichloroethane	62	64, 100, 98
2-Butanone	43**	57
1,1,1-Trichloroethane	97	99, 117, 119
Carbon Tetrachloride	117	119, 121
Bromodichloromethane	83	85
1,1,2,2-Tetrachloroethane	83	85, 131, 133, 166
1,2-Dichloropropene	75	77
Trichloroethene	130	95, 97, 132
Dibromochloromethane	129	208, 206
1,1,2-Trichloroethane	97	83, 85, 99, 132, 134
Benzene	78	---
cis-1,3-Dichloropropene	75	77
Bromoform	173	171, 175, 250, 252, 254, 256
2-Hexanone	43	58, 57, 100
4-Methyl-2-pentanone	43	58, 100
Tetrachloroethene	164	129, 131, 166
Toluene	91	92
Chlorobenzene	112	114
Ethylbenzene	106	91
Styrene	104	78, 103
Total Xylenes	106	91

* The primary ion should be used unless interferences are present, in which case, a secondary ion may be used.
** m/z 43 is used for quantitation of 2-butanone, but m/z 72 must be present for positive identification.

FIGURES

TestAmerica Connecticut Figure 1 128 Long Hill Cross Road
Shelton, Connecticut, 06484

QUALITY CONTROL APPROVAL REPORT

GC/MS Analysis Checklist QC Batch_____

Chain of Custody forms have been completed.		
Initial Calibration meets the following criteria: ( )ACOE ( )CLP4.1 ( )NYS95 ( )8260B ( )624 ( )524 rev4 ( ) T01/T02 ( ) other		
Continuing calibration meets the following criteria: ( )ACOE ( )CLP4.1 ( )NYS95 ( )8260B ( )624 ( )524 rev4 ( ) T01/T02 ( ) other		
Retention times and areas have been verified in the standards.		
VSUM criteria met for all accepted analysis: Internal Standards Surrogates - All accepted samples in clock		
Spike sublist selected and Form III generated (if required), reviewed for outliers		
QCS recovery results reviewed , copied and filed.		
All raw data is present with correct header information and quantitation factors. Areas have been verified. (Target Review)		
Prep Batch created on Labnet.		
OPC codes copied to batch and linked to data files.		
Sample tracking/breakdown sheets have been entirely updated for all batch results.		
All required TIC's have been generated. TICs have been checked for graphics and completion.		
Curve updated on Target.		
QC batch folder is complete per SOP.		
Batch guts copied and Labnet codes indicated for each job in batch.		
All samples qedited and chromatograms signed		
Manual integrations double signed and EICP printed.		
Corrective actions submitted (as needed).		

This data meets the requirements of the GC/MS SOP's, unless otherwise documented in a Corrective Action Report.

_____ Date_____
Authorizing Signature

DOC.# MSF00209.CT

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

Figure 1.1

TestAmerica CT

Quality Control Approval Report

Job #:_____

Client:_____

GC:_____ Volatiles:_____ Semivolatiles:_____ **Deliverables Requested:**_____

	Initial Approval Initials/Date	Final Approval Initials/Date
1. All samples & QC present	_____	_____
2. All raw data present and legible	_____	_____
3. Special Instructions identified	_____	_____
4. All required forms completed & Qualifiers verified	_____	_____
5. Injection Log copies present	_____	_____
6. IDL's are present (NYB).	_____	_____
7. NCMs in Lims	_____	_____
8. COC present (BNA/GC)	_____	_____
9. Standard Conc. form present (GC)	_____	_____
10. Reporting sheet updated	_____	_____
11. Method in LIMS Approved	____NA___	_____

Deliverables Feedback:

Final Approval:_____ **Date:**_____

GC/MS VOLATILES INJECTION LOG Instr. HP5972T

Standards Codes:	Routine Maintenance Performed:	Date:
BFB: IS:		QC Batch:
IU: CAL:		IDfile: Qcaled@
LCS: MS/MSD:		Calib file:
Other:		Method file:

Calibrated for the following (please check): Protocol= SW846( ) CLP ( ) NYSDEC( ) 524.2 ( ) Other( )
List= Tal( ) PP( ) 524.2 ( ) Other( )

Data file	CLP Data file	Client/Job#	Sample ID	Inj. Time	ALS #	DF	QF	Analyst	Comments	pH/WT

Witnessed by: Date:

Figure 2

Figure 3

Report Date: 07/10/2003

EXCEPTION REPORT

Method File: \\TARGET1_CT\Files\chem\VOA\mst.i\T030805.b\TAIRFULL.m
Analysis Type: WATER
Batch: \\TARGET1_CT\Files\chem\VOA\mst.i\T030805.b
Instrument ID: mst.i

Sample Name	Sample File	Run Date	IS Areas	Surr Rec	Over Range
VBLKT1	T0806.D	18-JUN-2003 11:31			
20ng QCS	T0807.D	18-JUN-2003 12:19			
203921-1	T0808.D	18-JUN-2003 13:21		H	
203921-2	T0809.D	18-JUN-2003 14:01			E
203921-3	T0810.D	18-JUN-2003 14:41			
203921-4	T0811.D	18-JUN-2003 15:21			E
203921-1	T0812.D	18-JUN-2003 16:01			E
203921-2	T0813.D	18-JUN-2003 16:41			
203921-3DUP	T0814.D	18-JUN-2003 17:21			
50ng-4BFB	TB163.D	18-JUN-2003 09:30			

FIGURE 4

NCM ID: 220-4474 **Date Opened:** 01/18/2008
NCM Type: Other - Observation **Date Closed:**
Lab Section: Volatiles GC/MS **Date Verified:**

Narrative
<<EXPLANATION REQUIRED>>

Affected Items

Description	Project Manager

Notifications

User Full Name	Date Received	Date	Notice Level	Verification Type
Decker, Larry	01/18/2008		Level 1	Review
Culik, Marsha	01/18/2008		Level 1	Review

Figure 5

Volatiles Tracking Sheet

Job No.__________________

Client:______________________

Due Date:______________

Verbal Due Date:_________

Deliverables:____________

TICs: Yes/No

Special Requirements:

mb /lcs	std code	Instument Batch	Prep	Analytical
/				
/				
/				
/				
/				
/				

Holding Time	Sample Number	Matrix	Protocol	Target List	Date run	Rerun(Y/N)	2nd Run	Data File	DL or RA Data File	Complete

FIGURE 6.0
GC/MS VOLATILE INSTRUMENT MAINTENANCE LOG

Date	Analyst	Problem	Maintenance Performed
1/6/99	KMR	IS. + Surr. Out of Range !	Cleaned Source

Date	Analyst	Problem	Maintenance Performed
1/6/99	D.H.	Tuning Repeller responses skewed	Disassembled + reassembled Source

Date	Analyst	Problem	Maintenance Performed
3/4/99	D.H.	Tune wouldn't pass	Cleaned source BFB- 0B314 good Batch 0φ181 1st Batch after cleaning

Date	Analyst	Problem	Maintenance Performed
3/8/99	D. Hubert	Troubleshooting IS problems IS Dropping off...	Put on new Tekmar 2,000 # 93033006 IS AREAS ARE CURRENTLY STABLE 02471-02474 VSTD050 3/9/99 BATCH 00190 03/09/99 LHD

Figure 7

Compound	m/z	RT(1)
Acetone	43	6.57
Methyl tert-Butyl Ether	73	8.16
1,1-Dichloroethene	96	6.83
trans-1,2-Dichloroethene	96	8.26
cis-1,2-Dichloroethene	96	10.24
1,1,1-Trichloroethane	97	11.48
Carbon Tetrachloride	117	12.10
cis-1,3-Dichloropropene	75	15.97
trans-1,3-Dichloropropene	75	17.17
4-Methyl-2-Pentanone	43	15.61
2-Hexanone	43	17.76
Ethylbenzene	106	20.58
Xylene (meta&para)	106	20.77
Xylene (ortho)	106	21.68
Propylbenzene	91	23.41
2-Chlorotoluene	91	23.56
4-Chlorotoluene	91	23.73
Butylbenzene	91	25.58
Isopropylbenzene	105	22.63
1,3,5-Trimethylbenzene	105	23.74
1,2,4-Trimethylbenzene	105	24.38
sec-Butylbenzene	105	24.58
tert-Butylbenzene	119	24.31
p-Isopropyltoluene	119	24.93
1,3-Dichlorobenzene	146	24.77
1,4-Dichlorobenzene	146	24.93
1,2-Dichlorobenzene	146	25.52
1,2,4-Trichlorobenzene	180	28.00
1,2,3-Trichlorobenzene	180	28.71

(1) The retention times listed will vary between instruments but the elution orders will not.

APPENDIX A

Initial Calibration:

- As per 8260B SOP plus:

  If the %RSD is below 15% for **each** compound in the initial calibration, then average RF can be used, if not, then linear regression is used for quantitation for those compounds above 15%. The minimum correlation coefficient for any compound using linear regression must also be 0.99. A copy of the linear regression plot from the calibration curve shall be included in the raw data. Samples can be run in the same initial calibration 12 hour BFB clock, without running a calibration check standard just an acceptable method blank and QCS is required.

Continuing Calibration:

As per 8260B SOP Criteria plus:

- ACOE projects must have all compound in the continuing calibration under 20%Difference. Compounds above 40%D will be noted in case narrative if calibration criteria is not met after normal corrective actions have been taken.

- Peak area must be within -50% to +100% of both the midpoint from the initial calibration and the continuing calibration.

- All TCL compounds must have an RRF of 0.05 or greater. Ketones 0.01 or greater.

Method Blank - As per requirements in Table 10.0. This may vary project to project.

LCS (020ppb_QCS) - All recoveries within laboratory generated guidelines. Up to four compounds can be out in the QCS. As per internal criteria set by laboratory.

- If a compound is out on the high side in the LCS and not detected in associated samples, then note in the case narrative and accept samples.

Full Matrix Spike / Full Matrix Spike Duplicate:

- Recoveries within laboratory generated guidelines. If compounds are outside of criteria and the LCS was acceptable, then accept FMS / FMSD. Note in case narrative that there is probable matrix interference.

MDL's

Soil MDL's are run as heated purge, Both water and medium level soils MDL's are run without heated purge.

Title: SOP for Total Cyanide
[Method SW846 9012B]

Approvals (Signature/Date):

Technical Manager Date	Health & Safety Manager / Coordinator Date
Quality Assurance Manager Date	Laboratory Director Date

This SOP was previously identified as SOP No. CVS05404.CT.

Any reference within this document to Severn Trent Laboratories, Inc. or STL, should be understood to refer to TestAmerica Laboratories, Inc. (formerly known as Severn Trent Laboratories, Inc.)

Facility Distribution No. _____ Distributed To:_____

1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 This method is used to determine the concentration of inorganic cyanide (CAS Registry Number 57-12-5) in wastes or leachate. The method detects inorganic cyanides that are present as either soluble salts or complexes. It is used to determine values for both total cyanide and cyanide amenable to chlorination. The reactive cyanide content of a waste is not distilled by this method (method 9012B). However this method can be used to quantify the concentration of cyanide from the reactivity test.

2.2 This method is applicable to the determination of cyanide in drinking, surface and saline waters, groundwaters, sediments and other solids, and domestic and industrial wastes.

2.3 This method covers the determination of cyanide by midi distillation with a semi-automated colorimetric analysis of the distillate.

2.4 The Lachat semi-automated spectrophotometric procedure is used for concentrations below 1 mg/L of cyanide. The working range for the Lachat auto-analyzer is 10 ug/L (0.010 mg/L) to 300 ug/L (0.300 mg/L). Higher level samples must be diluted to fall within this range.

2.5 The detection limit for soils is dependent on the percent moisture of the sample.

2.6 The document control number for this SOP is CT-CVS-54, rev 5.

2.7 This method can also be used for the following methods: 335.2, 335.4, and 4500CN.

2.8 The Army Corps of Engineers has method specific requirements which are listed in Appendix B.

3.0 TERMS AND DEFINITIONS

3.1 Refer to the SOP for Laboratory Term and Definitions.

4.0 SUMMARY OF METHOD

4.1 **Method Deviations:**

4.1.1 Midi reflux-distillation method is used instead of the macro distillation setup. This was done to minimize waste.

4.1.2 Working standards recipe is different. The 1:1 dilution step is omitted to minimize waste.

4.1.3 Reporting limit for cyanides is 10 ug/L instead of 20 ug/L.

4.2 The cyanide as hydrocyanic acid (HCN), is released from cyanide containing samples by means of a midi reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined colorimetrically.

4.3 In the colorimetric measurement, the cyanide is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at pH less than 8 without hydrolysis to the cyanate. After the reaction is complete, color is formed on the addition of pyridinebarbituric acid reagent. The absorbance is read at 570 nm. To obtain colors of comparable intensity, it is essential to have the same salt content in both the samples and the standards.

4.4 This method is based on method 9012B, SW-846.

5.0 INTERFERENCES

5.1 Interferences are eliminated or reduced by using the distillation procedure. Chlorine and sulfide are interferences in this method.

5.2 Oxidizing agents such as chlorine decompose most cyanides. Chlorine interferences are removed by adding an excess of ascorbic acid to the waste prior to preservation and storage. The ascorbic acid reduces the chlorine to chloride, which does not interfere.

5.3 Sulfide interference is removed by adding an excess of bismuth nitrate to the waste (to precipitate the sulfide) before distillation.

5.4 Nitrate and/or nitrite may cause high cyanide results. During the distillation, nitrate and nitrite will form nitrous acid, which will react with some organic compounds to form oximes. These compounds once formed will decompose under the test conditions to produce HCN. The possibility of interference of nitrate and nitrite is eliminated by pretreatment with sulfamic acid just before distillation. Nitrate and nitrites are interferences when present at levels higher than 10 mg/L and in conjunction with certain organic compounds.

5.5 Thiocyanate is reported to be an interference when present at very high levels. Levels of 10 mg/L were not found to interfere in method 9010.

5.6 The presence of surfactants, detergents, fatty acids, and other organic compounds may cause the sample to foam during refluxing. If this occurs, the addition of an agent such as Dow Corning 544 antifoaming agent will prevent the foam from collecting in the condenser.

6.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

6.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Potassium Cyanide will give off Hydrogen Cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.

6.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Aci	Corrosive Oxidizer Dehydra-dat	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Chlora-mine T Hydrate	Poison		May be harmful by inhalation, ingestion, or skin absorption. This material is irritating to mucous membranes and upper respiratory tract. Avoid conta and inhalation.

Pyridine	Flamma-ble Irritant	5 ppm-TW/	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Sympton include redness and severe pain. Absorption throug the skin may occur, resulting in toxic effects similar t inhalation. May act as a photosensitizer. Vapors cau eye irritation. Splashes cause severe irritation, possib corneal burns and eye damage.
Potassium Cyanide	Poison Corrosive	5 Mg/M3 TWA as CN	**This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death.** Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heart beat, unconsciousness, convulsions, coma and death Solutions are corrosive to the skin and eyes, and ma cause deep ulcers, which heal slowly. May be absorb through the skin, with symptoms similar to those no for inhalation. Symptoms may include redness, pain blurred vision, and eye damage.
Silver Nitrat	Corrosive Poison	0.01 mg/m^3 (TWA)	This is a corrosive, poisonous material. It will cause burns to any area of contact and is harmful if inhale Ingestion may cause death. Contact with other mate may cause fire.
1 - Always add acid to water to prevent violent reactions.			
2 - Exposure limit refers to the OSHA regulatory exposure limit.			

6.3 At a minimum, wear PVC gloves, a lab coat, and eye protection when handling samples and reagents.

6.4 Pyridine and concentrated hydrochloric acid must be used under a fume hood.

6.5 Potassium cyanide (KCN) is highly toxic; avoid inhalation of dust or contact with the solid or solutions. Avoid contact with acid.

6.6 Samples are distilled under a laboratory fume hood.

7.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

7.1 Samples are collected in plastic bottles, stored at 4°C (±2°C) and transported to the laboratory as soon as possible. Sample bottles are never reused.

7.2 Oxidizing agents such as chlorine decompose most cyanides. To determine whether oxidizing agents are present, test a drop of the sample with potassium iodide-starch test paper. A blue color indicates the need for treatment. Add a few crystals of ascorbic acid at a time until a drop of sample produces no color on the indicator paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume.

7.3 Water samples are preserved with sodium or potassium hydroxide until the pH ≥ 12 at time of collection.

7.4 Samples should be chilled to 4°C.

7.5 When samples are properly preserved, cyanide samples can be stored for up to 14 days prior to sample preparation steps (12 days for NYSDEC samples).

7.6 Soil samples are not dried prior to analysis; a separate percent solids determination must be made.

7.7 Samples must be distilled and analyzed within 14 days from time of sampling.

8.0 APPARATUS AND MATERIALS

8.1 Andrews Glass Model 110-10 R Midi cyanide distillation system.

8.2 Lachat QuikChem 8000 Automated Flow Injection Analyzer, which includes:

8.2.1 Lachat XYZ Automatic Sampler, Model 2100-000

8.2.2 Lachat Proportioning pump, Model 2200-010

8.2.3 Cyanide manifold, reaction module 10-204-00-1-A

8.2.4 Injection module with a 150 cm, 0.8 mm i.d. sample loop

8.2.5 Colorimeter

8.2.5.1 Flow cell, 10 mm, 80 uL

8.2.5.2 Interference filter wavelength, 570 nm

8.2.6 Heating block with temperature controller: (Set at 60°C.)

8.2.7 QuikChem 8000 Software System

8.3 Printer

8.4 Vacuum source.

8.5 Class A (pipette and volumetric flasks)

8.6 Analytical balance, Denver Instrument Model P-214 or equivalent

8.7 Toploading balance, Denver Instrument Model P-602 or equivalent

9.0 REAGENTS AND STANDARD PREPARATION

Note: the expiration dates for chemicals used in this analysis are twenty years from time of receipt for raw, solid materials and one year from time of receipt for raw, liquid materials except where noted. Reagents made from raw materials are good for one year except where noted.

9.1 Distillation and Preparation Reagents

9.1.1 Reagent water - (ASTM Type II) deionized water.

9.1.2 Sodium hydroxide solution, 50%: Dissolve 500g of NaOH in reagent water and dilute to 500 mL.

9.1.3 Sodium hydroxide solution, 1.25N: Dissolve 50g of NaOH in reagent water and dilute to 1 liter.

9.1.4 Sodium hydroxide solution, 0.25N: Dissolve 10g of NaOH in reagent water and dilute to 1 liter.

9.1.5 Magnesium chloride solution, 51% (w/v): Dissolve 510g of $MgCl_2 \bullet 6H_2O$ in reagent water and dilute to 1 liter.

9.1.6 Sulfuric acid, 50% (v/v): Carefully add a portion of concentrated H_2SO_4 to an equal portion of reagent water. Caution: the solution will get extremely hot. Do not add water to concentrated acid.

9.1.7 Ascorbic acid: crystals.

9.1.8 Bismuth nitrate (.062M), $Bi(NO)_3 \bullet 5H_2O$: Dissolve 30 g), $Bi(NO)_3 \bullet 5H_2O$ in 100 mL of water. While stirring, add 250 mL of glacial acetic acid, CH_3COOH. Stir until dissolved and dilute to 1 liter with water.

9.1.9 Sulfamic acid (0.4N), H_2NSO_3H: Dissolve 40 g H_2NSO_3H in 1 liter of water.

9.1.10 KI-starch paper.

9.1.11 Lead acetate paper.

9.2 **Standards**

9.2.1 Stock cyanide solution, 1000 mg/L CN: Dissolve 2.51g of KCN and 2.0g KOH in reagent water and dilute to 1 liter. Standardize with 0.0192N $AgNO_3$ at time of preparation. The solution is stable for six months and shall be stored at 4°C.

9.2.2 Standard cyanide solution, 1mL = 100ug: Transfer 20mL of 1.25N NaOH (8.1.3) to a 100mL volumetric flask. Using a 25mL burette, transfer the amount of cyanide stock solution (9.2.1) indicated on the bottle to the flask. Dilute to the mark with reagent water. Prepare fresh weekly.

9.2.3 Standard cyanide solution, 1mL = 5ug: Transfer 20mL of 1.25N NaOH (8.1.3) to a 100mL volumetric flask. Using a Class A pipet, transfer 5mL of standard cyanide solution (9.2.2) to the flask and dilute to the mark with reagent water. Prepare fresh weekly.

9.2.4 Standard cyanide solution, 1mL = 1ug: Transfer 40mL of 1.25N NaOH (8.1.3) to a 200mL volumetric flask. Using a Class A pipet, transfer 2mL of standard cyanide solution (9.2.2) to the flask and dilute to the mark with reagent water. Prepare fresh daily.

9.2.5 Rhodanine indicator: Dissolve 20mg of [p-(dimethylamino)-benzylidene]-rhodanine in 100mL acetone.

9.2.6 Silver nitrate solution, 0.0192N: Prepare by crushing approximately 5g $AgNO_3$ crystals and drying to a constant weight at 104°C. Weigh out 3.2647g of dried $AgNO_3$ and dissolve in reagent water. (Note: the Denver Instrument, P-214 analytical balance or equivalent is used to weigh out the silver nitrate.) Dilute to 1 liter (1mL = 1mg CN). Standardize against 0.0141N NaCl.

9.2.7 Potassium chromate indicator solution: Dissolve 50g K_2CrO_4 in sufficient reagent water. Add silver nitrate solution until a definite red precipitate is formed. Let stand for at least 12 hours, filter, and dilute to 1 liter with reagent water.

9.2.8 Primary standard sodium chloride, 0.0141N: Dissolve 824.1mg NaCl (NBS-dried 20 minutes at 104°C) in reagent water and dilute to 1 liter.

9.2.9 ICV Stock cyanide solution, 1000 mg/L CN: Dissolve 2.51g of KCN and 2.0g KOH in reagent water and dilute to 1 liter. Standardize with 0.0192N $AgNO_3$ at time of preparation. The solution is stable for six months and shall be stored at 4°C.

NOTE: Make sure the source of the KCN used for the ICV and the cyanide stock solution are different.

9.2.10 ICV Standard cyanide solution, 1mL = 100ug: Transfer 20mL of 1.25N NaOH (9.1.3) to a 100mL volumetric flask. Using a 25mL buret, transfer the amount of ICV cyanide stock solution (9.2.9) indicated on the bottle to the flask. Dilute to the mark with reagent water. Prepare fresh weekly.

9.2.11 ICV Standard cyanide solution, 1mL = 1.5ug: Transfer 40mL of 1.25N NaOH (9.1.3) to a 200mL volumetric flask. Using a Class A pipet, transfer 3mL of ICV standard cyanide solution (9.2.10) to the flask and dilute to the mark with reagent water. Prepare fresh daily.

9.3 **Semi-Automated Spectrophotometric Reagents**

9.3.1 Phosphate buffer solution, 1M: Dissolve 97g of KH_2PO_4 H_2O in reagent water and dilute to 1 liter. Store at 4°C.

9.3.2 Chloramine-T solution, 0.4% (w/v): Dissolve 2.00g of chloramine-T in reagent water and dilute to 500mL. Prepare fresh weekly.

9.3.3 Pyridine barbituric acid color reagent solution: Prepare this solution in the hood. Transfer 15g of barbituric acid to a 1 liter volumetric flask and add about 100mL of reagent water, rinsing down the sides of the flask to wet the barbituric acid. While stirring, add 75mL of pyridine and 15mL of concentrated HCl. Mix until all the barbituric acid is dissolved. Dilute to 1 liter with reagent water and store at 4°C. This reagent is stable for approximately six months if stored in a cool, dark place.

9.3.4 Carrier, 0.25N NaOH: Dissolve 10g of NaOH in 1 liter of reagent water.

10.0 CALIBRATION

10.1 Calibration Standards

TABLE I. Calibration Standards

Concentration (ug/L CN)	mL of Standard Cyanide Soln., 1mL = 5ug (9.2.3)	Final Volume, mL*
300	15	250
200	10	250
100**	5	250
50.0	2.5	250

20.0	1.0	250
10.0	0.5	250

*Bring to volume with 0.25N NaOH (9.1.4)
**Check Standard (CCV)
NOTE: The Calibration Blank, ICB, and CCB's consist of 0.25N NaOH (9.1.4)

10.2 Instrument Calibration

The spectrophotometer must be calibrated each time the instrument is set up. The instrument standardization date and time shall be included in the raw data. Calibration standards must be prepared fresh each time an analysis is to be made and discarded after use. Prepare a blank and the standards indicated in the table above. NOTE: One calibration standard must be at the Reporting Limit, which is 10.0 ug/L. Standards must bracket the concentration of the samples. The correlation coefficient for the curve must be ≥ 0.995. Two additional standards (300 ug/L and 40.0 ug/L standard) must be distilled and compared to similar values on the curve to ensure that the distillation technique is reliable. Acceptable range for distilled standards is $\pm$ 10%.

10.3 Balance Calibration

The top loading balance used in weighing stock reagents, percent solids, and soil samples is calibrated daily by comparing observed values with known values of 0.50, 5.00, and 50.00g Class S weights. (Class S weights are recalibrated every five years by an outside vendor.)

The calibration is performed in the following manner:

Depress the "Tare" bar on the balance to "zero" the instrument. When the zero (0.00g) signal is stable for 10 seconds or more, place a standard weight onto the pan. Allow the reading to stabilize. When stable for 10 seconds or more, record the reading.

Note: The acceptance criteria for Class S weights used to calibrate the top loading or analytical balance should be within $\pm$ 0.1 % or 0.5 mg, whichever is greater.

11.0 QUALITY CONTROL

11.1 Initial Calibration Verification (ICV)

Immediately after the Lachat QuikChem AE Flow Injection Analyzer has been calibrated, the accuracy of the initial calibration shall be verified and documented by the analysis of the Initial Verification Solution for cyanide. This solution must be distilled the same way as the samples. Acceptable recovery for the ICV is $\pm$ 10% of the ICV

ICV true value.

If the Initial Verification Solution is not available from the EPA, or where a certified solution for cyanide is not available from any source, analysis shall be conducted on an independent standard at a concentration other than that used for instrument calibration but within the calibration range. An independent standard is defined as a standard composed of the analyte from a different source than that used in the standards for the instrument calibration.

An ICV must be distilled with each batch of samples analyzed; the samples distilled with an ICV must be analyzed with that particular ICV. For aqueous CN samples, the ICV for CN also serves as the Laboratory Control Sample (LCS). A separate LCS is required for soil CN samples.

11.2 Continuing Calibration Verification (CCV)

To ensure calibration accuracy during each run, a mid-range standard must be prepared to be used for continuing calibration verification and must be analyzed at a frequency of 10% of samples or every two hours during an analysis run, whichever is more frequent. The standard must be analyzed at the beginning of the run, after ten samples, and after the last analytical sample. The same continuing calibration standard must be use throughout the analysis run for a case of samples received. Acceptable recovery for the CCV is ± 10 % of the true value.

11.3 Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Preparation Blank (PB) Analyses

A calibration blank must be analyzed for cyanide at the beginning of the run, after every initial and continuing calibration verification, at a frequency of 10 percent of samples or every two hours, whichever is more frequent, and after the last analytical sample.

At least one method (or reagent) blank consisting of reagent water processed through the distillation procedure must be prepared and analyzed with each sample batch of 20 samples or less digested.

The values for the method blank must be recorded in mg/L for aqueous samples and in mg/Kg for solid samples. The concentration of the blank must be less than one-half the reporting limit.

11.4 Spike Sample Analysis

The spike sample analysis is designed to provide information about the effect of the sample matrix on the measurement methodology. The spike is added prior to any

distillation steps. At least one spike must be performed on each group of samples of a similar matrix (i.e. water, soil) or every 20 samples or fewer. Samples identified as field blanks cannot be used for spike sample analysis.

The analyte spike must be added in the concentration of 40 ug/L for both water and soil matrices. The spike amount added is based on the final distillate volume. For example, the midi-distillation procedure requires the addition of 2.0 ug cyanide to the sample prior to distillation (based on the final distillate volume of 50mL). For soil samples, 2.0 ug cyanide is added prior to distillation regardless of the amount of sample used for distillation. Acceptable recovery for spikes is ± 25 % of spike added.

11.5 Duplicate Sample Analysis

One duplicate sample shall be analyzed from each group of samples of a similar matrix type (i.e. water, soil) for each batch of 20 or fewer samples.

11.6 Laboratory Control Sample (LCS) Analysis

Solid Laboratory Control Samples (LCS) must be analyzed for cyanide using the same sample preparations and analytical method. The acceptable range for solid LCS is defined by the upper and lower limits established by the manufacturer.

Percent solids determination is not required on the LCS. One solid LCS must be prepared and analyzed for each batch of 20 samples distilled.

11.7 Method Detection Limit (MDL) Determination

Every year, the instrument detection limits (in mg/L) shall be determined. The MDL will be calculated by multiplying by 3.14, the standard deviations obtained on the analysis of a standard solution at a concentration 3x-5x the IDL, with seven consecutive replicates.

11.8 Summary Of Quality Assurance/Quality Control

Instrument Calibration - Calibration Standards in graduated amounts, one of which must be at the low reporting level (non-distilled), and Calibration Blank; include date of analysis.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) - Analysis of distilled known standard (ICV) then a mid-range standard (non-distilled) (CCV) must be analyzed. The CCV is then analyzed after every tenth sample and after the last analytical sample.

Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Method Blank (MB) - Calibration Blank (non-distilled) must be analyzed at the beginning of the run and after every initial and continuing calibration verification. Method Blank - consisting of reagent water processed through the distillation procedure must be analyzed with each batch of samples.

Distilled Standards: a 40.0 ug/L and 300 ug/L standard must be distilled with the samples per day.

Spiked Sample - One sample spike must be performed at a frequency 20% of samples of a similar matrix. Field blanks cannot be used for spike analysis.

Duplicate Sample Analysis - One duplicate must be performed on each batch of 20 samples of a similar matrix. Field blanks cannot be used as duplicates.

Laboratory Control Sample (LCS) - Solid Laboratory Control Samples must be prepared and analyzed for every batch of solid samples analyzed.

11.9 Instrument Maintenance

An instrument maintenance log is kept for the Lachat Autoanalyzer.

11.10 All stock solutions, working standards, LCS's, and reagents must be entered into LIMS.

12.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

12.1 Midi-distillation Procedure

12.1.1 This procedure utilizes an Andrews Glass Co., Model 110-10R midi cyanide distillation system and requires a sample aliquot of 50mL or less for aqueous samples and one gram for solids. NOTE: All samples must initially be run undiluted (i.e. aqueous samples must first be run with a 50mL aliquot and solid samples using a one gram sample).

When the cyanide concentration exceeds the highest calibration standard, appropriate dilution (but not below the reporting limit) and reanalysis of the sample is required.

12.1.2 Sample control tests aqueous samples with pH paper to ensure pH > 12 when the samples arrive.

12.1.3 Test samples for chlorine prior to distillation: transfer an aliquot of soil sample to a test tube (add approximately 10mL of reagent water to each soil test tube). Test a drop of sample with potassium iodide starch test paper. A blue color indicates the presence of chlorine. If chlorine is present, add ascorbic acid crystals to the sample at

a time until a drop of the sample produces no color on the indicator paper. Then add an additional 0.6 g ascorbic acid for each liter of sample prior to distillation. Record whether chlorine is present or is not.

12.1.4 Test samples for sulfide prior to distillation: transfer an aliquot of soil sample to a test tube (add approximately 10mL of 0.25N NaOH to each soil test tube). Test a drop of sample with lead acetate paper. A black color indicates the presence of sulfide. If the sulfide test is positive, add 5 mL of 0.062M bismuth nitrate through the air inlet tube. Mix for three minutes.

NOTE: For samples with sulfide, all standards must be distilled in the same manner as the samples using the method of standard additions (for example, bismuth nitrate must also be added to standards). Standards distilled in this way will give a linear curve, at low concentrations, but as the concentration increases, the recovery decreases. It is recommended that at least five standards be distilled.

**** Distill same concentration of standards used for calibration for samples without sulfides.**

12.1.5 For aqueous samples: Add 50mL of sample, or an aliquot diluted to 50mL, into the reflux flask along with 2 or 3 boiling chips.

12.1.6 For solid samples: Weigh 1-2 g of sample (record to the nearest 0.01g) into the reflux flask and dilute to 50mL with distilled water. Add 2 or 3 boiling chips.

12.1.7 Add 50 mL of 0.25N NaOH into a clean absorber flask.

12.1.8 Beginning with the left most heat block hole, connect the reflux flask, absorbing flask, and condenser in the train. Up to ten samples may be loaded per heat block at one time.

12.1.9 Turn on the chiller for the condenser water; the flow should be at 60 GPH for each block.

12.1.10 Turn on the vacuum and adjust each valve to give a flow of three bubbles per second in each reflux flask.

12.1.11 If samples are known or suspected to contain nitrate or nitrite, or bismuth nitrate was added to the sample, add 5 mL of 0.4N sulfamic acid solution through the air inlet tube. Mix for three minutes.

NOTE: Excessive use of sulfamic acid could create a method bias.

12.1.12 Slowly inject 5mL of 50% (v/v) H_2SO_4 through the air inlet tube of each reflux impinger. Allow to mix for five minutes. NOTE: The acid volume must be sufficient to bring the

to bring the sample/solution pH to below 2.0.

12.1.13 Inject 2mL of magnesium chloride solution through the air inlet of each reflux impinger. Inject another 2mL if excessive foaming occurs.

12.1.14 Turn on the heat block, pushing the red rocker switch towards the rear of the unit. (Light will glow.) The heat block is factory preset to 125°C. Turn on the timer, setting for 75 minutes). The green timer light and amber heat lights will glow. This time setting allows for 15 minutes heat up time (may vary relative to location and hood conditions) and 60 minutes of reflux time. As the solution heats to boiling, it may be necessary to adjust the vacuum flow to prevent solution backup.

12.1.15 The timer will automatically turn off the heat block after the selected time has expired. (The vacuum and condenser water are unaffected.) Allow to cool for 15 minutes before turning off the vacuum and condenser water.

12.1.16 After cooling, close each vacuum valve and disconnect the slip connector between the absorber flask and reflux flask. Starting from the right, open the vacuum valve to draw all of the solution from the absorbing impinger frit. Close the vacuum valve and remove the impinger frit. Disconnect the absorber flask to vacuum tubing and remove the absorber impinger.

12.1.17 Seal the receiving solutions and store at 4°C until time of analysis. The solutions must be analyzed for cyanide within the 14 days from time of sampling.

12.2 Semi-Automated Spectrophotometric Determination

12.2.1 Inspect modules for proper connections.

12.2.2 Turn on power and all modules. Allow the heating block to warm up to 60°C.

12.2.3 Degas reagents for one minute. Place reagent feedlines into the proper containers. Raise the tension levers on the pump tube cassettes.

12.2.4 Pump system until a stable baseline is established.

12.2.5 Program the data system with the sample tray information (follow the order of development specified in Appendix A.)

12.2.6 Place calibration standards and blank in sample tray in descending order of concentration followed by the samples submitted for analysis. Note: calibration blanks consist of 0.25N NaOH.

12.2.7 As results are generated, refer to the Acceptance Of Data section (14.0) to check if criteria

criteria are being met. Samples must be run undiluted first. If a sample needs to be diluted, use 0.25N NaOH to make up the dilution.

12.2.8 At the end of the run, place all feedlines in water, flush the system, and pump dry. Turn off the pump, all modules, and chart recorder; release the tension on the pump tube cassettes.

13.0 CALCULATIONS

13.1 Cyanide Concentration in Samples:

13.1.1 Calculate the cyanide of aqueous samples in mg/L of original sample, as follows:

$$\text{CN, mg/L} = \frac{A \times D \times F}{B}$$

where: A = ug/L CN of sample from Lachat print-out
B = Liters of original sample for distillation (0.050 L) (See 12.1.4)
D = any dilution factor necessary to bracket sample value within standard values
F = sample receiving solution volume (0.050 L)
The minimum value that can be substituted for A is 10 ug/L.

13.1.2 Calculate the cyanide of solid samples in mg/Kg of original sample, as follows: (A separate determination of percent solids is performed by the Metals Department and is accessed when entering results onto LIMS.)

The concentration of cyanide in the sample is determined as follows:

$$\text{CN, mg/Kg} = \frac{A \times D \times F}{B \times E}$$

where: A = ug/L CN of sample from Lachat print-out
B = wet weight of original sample in g (See 12.1.5)
D = any dilution factor necessary to bracket sample value within standard values
E = % solids/100
F = sample receiving solution volume (0.050 L)

The minimum value that can be substituted for A is 10 ug/L.

13.2 Percent Recovery

13.2.1 Spike recoveries are calculated as follows:

$$\% \text{ Recovery} = \frac{(SSR - SR) \times 100}{SA}$$

where: SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

When sample concentration is less than the instrument detection limit, use SR = 0 for calculating percent recovery.

13.2.2 Percent Recovery of Initial and Continuing Calibration Verification

For Initial Calibration Verification (ICV):

$$\% \text{ Recovery} = \frac{\text{Found (ICV)} \times 100}{\text{True (ICV)}}$$

For Continuing Calibration Verification (CCV):

$$\% \text{ Recovery} = \frac{\text{Found (CCV)} \times 100}{\text{True (CCV)}}$$

13.2.3 Percent Recovery of Solid LCS

$$\% \text{ Recovery} = \frac{\text{Solid LCS Found} \times 100}{\text{Solid LCS True}}$$

If the analyte concentration is less than the IDL, a value of zero shall be substituted for the Solid LCS Found.

The acceptable range for the solid LCS is defined by the lower and upper limits established by the manufacturer.

13.3 Relative Percent Difference

The Relative Percent Difference is calculated as follows:

$$RPD = \frac{[S - D] \times 100}{(S + D)/2}$$

where: S = Sample result

D = Duplicate result

A value of zero shall be substituted for S or D if the analyte concentration is less than than the LDL in either one.

For solid samples, the concentration of the original sample shall be computed using the weight and % solids of the original sample. The concentration of the duplicate sample shall be computed using the weight of the duplicate sample but the % solids of the original sample.

14.0 ACCEPTANCE OF DATA

14.1 Initial Calibration Verification (ICV): The control limit for the ICV is ± 10 percent of true value.

Action on failure: When measurements exceed the control limit, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration verified.

14.2 Continuing Calibration Verification (CCV): The control limit for the CCV is ± 10 percent of true value.

Action on failure: If the deviation of the CCV is greater than the control limit, the instrument must be recalibrated, the calibration verified, and the preceding ten samples analyzed since the last compliant calibration verification must be reanalyzed.

14.3 Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Method Blank (MB): The absolute value of the concentration in the blank (ICB, CCB) must be less than or equal to the half the reporting limit.

Action on failure: If the magnitude (absolute value) of the calibration blank result exceeds the half the reporting limit, terminate analysis, correct the problem, recalibrate, verify the calibration, and reanalyze the preceding ten analytical samples or all analytical samples analyzed since the last compliant calibration blank.

The method blank result is reported for each batch and is used to ascertain whether sample concentrations reflect contamination in the following manner:

1) If the absolute value of the concentration of the blank is less than one-half the reporting limit, no correction of sample results is performed.

2) If the cyanide concentration in the blank is above the reporting limit, the lowest concentration of cyanide in the associated samples must be 10x the blank concentration. Otherwise, all samples associated with the blank with a cyanide

concentration less than 10x the blank concentration and above the reporting limit must be redistilled and reanalyzed (except for an aqueous soil field blank). The sample concentration is not to be corrected for the blank value.

14.4 Spike Sample Recovery: The acceptable range for spike recovery is 75-125 percent; an exception to this is when the sample concentration exceeds the spike level by a factor of four or more.

Action on failure: When the pre-distillation spike recovery falls outside the control limits and the sample result does not exceed 4x the spike added, a post-distillation spike must be performed and the method of standard additions will be performed on all samples that suffer from matrix interferences such as samples that contain sulfides.

14.5 Duplicate Sample Analysis: A control limit of $\leq$20% for RPD shall be used for original and duplicate samples greater than or equal to 5x's the Reporting Limit. If both samples are less than 5x's the RL, the difference between the two results must be less than the RL.

If both sample values are less than the reporting limit, the RPD is not calculated.

Action on failure: If the duplicate results are outside the control limits, reanalyze the sample and duplicate.

14.6 METHOD OF STANDARD ADDITION

The standard of method addition involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analytical signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift.

14.6.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the same sample solution, each of volume Vx, are taken. To the first (labeled A) is added a known volume Vs of a standard cyanide solution of concentration Cs. To the second aliquot (labeled B) is added the same volume Vs of the 0.25 N NaOH solution. Then the two aliquots are distilled and analysed.

The unkown sample concentration Cx is calculated:

$$Cx = \frac{S_B VsCs}{(S_A - S_B)V_X}$$

where S_A and S_B are the concentration of solution A and B, respectively. Care should be taken such that S_A is roughly twice S_B on the average.

14.7 Distilled Standards: 40.0 ug/L range and 300 ug/L range control limit is ± 10% of the true value.

Action upon failure: Analyst should find the cause before proceeding. And all samples associated with the failed standards must be redistilled and reanalyzed.

14.8 Solid LCS: The control limits for the cyanide solid LCS are defined by the lower and upper limit values established by the manufacturer.

Action on failure: If the results for the solid LCS fall outside the control limit, the analysis must be terminated, the problem corrected, and the samples associated with that LCS redistilled and reanalyzed.

15.0 REPORTING OF RESULTS

15.1 Results are reported by importing data into Lims and worked up into reportable form.

15.2 NCM's should include any holding time problems, QC failures and any other problems associated with sample analysis.

15.3 The practical quantitation limit (PQL) for cyanide is 10.0 ug/L for aqueous samples. For soils, the nominal PQL is 0.50 mg/Kg, however, sample PQLs must be calculated to take into account the actual sample weight and percent solids. Two significant figures are reported for results less than 10.0 ug/L, and three significant figures for results equal to or greater than 10.0 ug/L.

16.0 POLLUTION PREVENTION

16.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

16.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

16.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

16.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

16.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

16.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

17.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

17.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Wastewater containing 1.5% Pyridine (Employees must dispose of pyridine waste in the drum specifically labeled for pyridine waste in the waste disposal area.) **A faceshield must be worn when dumping pyridine waste into the drum in the Hazardous Waste room.**

17.2 All waste shall be managed in accordance with all state and federal requirements. See the Testamerica CT Hazardous Waste Management Plan.

17.3 All personnel who handle or generate waste must be trained within six months of employment in proper waste handling and requirements.

18.0 SUPPLEMENTAL DOCUMENTS

18.1 Method 9012B SW-846, Revision 2, November 2004.

19.0 REFERENCES

19.1 Method 9012B SW-846, Revision 2, November 2004.

19.2 Methods Manual for the QuikChem Automated Ion Analyzer, Lachat Instruments, Mequon, WI, revised February 1989.

19.3 Instruction Manual, "Midi-Dist," Model 110-10R Midi Cyanide Distillation System, Andrews Glass Co., Vineland, NJ.

19.4 Desk Audit Report, U.S. Army Corps of Engineers, HTRW Center of Expertise, Omaha, Nebraska, December, 2003.

20.0 SUBSTANTIVE REVISIONS

20.1 Original document, 10/01/99.

20.2 Added 20.0 ug/L calibration standard, 05/16/02.

20.3 Section 2.7 added, 05/16/02.

20.4 Section 7.5 revised to include NYSDEC holding time, 05/16/02.

20.5 Sections 11.10 and 15.1 revised to reflect Labnet reporting system, 05/16/02.

20.6 Page 2-Health and Safety Officer signature requirement added, 01/13/05.

20.7 Section 2.8 added to refer to Appendix B (USACE requirements), 01/13/05.

20.8 Sections 6.1 and 6.2 added in Safety, 01/13/05.

20.9 Section 8.2.1-added Model numbers, 01/13/05.

20.10 Sections 8.6 and 8.7-added balances used including model numbers, 01/13/05.

20.11 Section 9.0 revised to include shelf-life of reagents and solid and liquid chemicals used, 01/13/05.

20.12 Section 9.2.6 added balance used to weigh silver nitrate per USACE requirement, 01/13/05.

20.13 Sections 11.1, 11.2, 14.1, 14.2-changed acceptance criteria for ICV and CCV to ± 10 %, 01/13/05.

20.14 Section11.3 revised acceptance criteria for Method Blank to < ½ the RL, 01/13/05.

20.15 Section 12.1.6 revised weight required for soil samples as "1.00 ± 0.05g," 01/13/05.

20.16 Section 12.1.9 added reference to chiller; changed flow to 60 GPH per block, 01/13/05.

20.17 Section 12.1.14 revised heating time to 75 minutes, 01/13/05.

20.18 Section 12.2.5 changed Appendix III to Appendix A, 01/13/05.

20.19 Section 12.2.7 removed reference to Appendix IV, 01/13/05.

20.20 Section 13.3 substituted an absolute sign for the parentheses in the numerator for RPD calculation per USACE request, 01/13/05.

20.21 Section 14.3 substituted "Method Blank" for "Preparation Blank;" changed acceptance criteria to <1/2 RL, 01/13/05.

20.22 Section 14.5 revised RPD control limit from 20% to ≤ 20%, 01/13/05.

20.23 Sections 17.0 and 17.1 revised Waste Management Section per STL requirements, 01/13/05.

20.24 Section 19.4 added reference to USACE, 01/13/05.

20.25 Appendix B added for USACE method specific requirements, 01/13/05.

20.26 Added (7) to Appendix B to address USACE requirements for analysis of a low concentration standard, 04/28/05.

20.27 Changed reference from 9012A to 9012B, 05/03/07.

20.28 Section 6.2 added silver nitrate to Primary Materials chart.

20.29 Sections 8.6 and 9.2.6 changed analytical balance to Denver Instrument Model P-214.

20.30 Section 8.7 changed toploading balance to Denver Instrument Model P-602.

20.31 Section 10.3 revised calibration of Class S weights to every five years.

20.32 Changed sections 11.10, 13.1.2, and 15.1 to reference LIMS instead of Labnet.

20.33 Section 15.2 changed "Corrective Action" reference to "NCM."

20.34 Section 18.1 and 19.1 revised to reference method 9012B, Revision 2, November, 2004.

20.35 Section 2.7 removed reference to 335.1.

20.36 Section 17.1 modified to include use of faceshield when dumping pyridine waste.

20.37 Added new TestAmerica SOP header and control number, changed name, 01/16/08.

APPENDIX A

LACHAT QUIKCHEM AUTOMATED FLOW INJECTION ANALYZER: ORDER FOR LOADING SAMLE TRAY

Curve (Instrument Calibration Standards):

Cup	Standard (ug CN/L)
A	300
B	200
C	100
D	50.0
E	20.0
F	10.0
G	0.0

Sample Tray:

Cup	**Parameter**
1	ICV1
2	ICB1
3	CCV1
4	CCB1
5	S40
6	S300
7	LCS (for soils)
8	MB
9-14	Samples
15	CCV2
16	CCB2
17-26	Samples
27	CCV3
28	CCB3
29-38	Samples
39	CCV4
40	CCB4
Etc.,	

APPENDIX B

USACE REQUIREMENTS FOR CYANIDE

The USACE has method specific requirements for cyanide analysis which are listed below:

(1) The acceptance criteria for the Method Blank is less than one-half the RL; cyanide concentrations for ICB's and CCB's should be less than the MDL.

(2) The Chloramine-T solution should be prepared fresh daily.

(3) The shelf-life of pyridine-barbituric acid is six months if stored in a cool dark place; if not, one month .

(4) The second source ICV should be undistilled.

(5) For all samples with results greater than the PQL, the acceptance criteria for RPD is $\leq$ 20%.

(6) An LCS from the same source as the initial calibration standards, at the concentration of concern, if known, or mid-level, and distilled should be analyzed per day.

(7) The low concentration distilled standard should be at 10 ug/L instead of 40 ug/L; the recovery of the low concentration distilled standard should be with ±15% of true value; the recovery of the high concentration distilled standard (300 ug/L) should be within ±10% of true value.

Title: SOP for TCLP Preparation
[Method SW846 1311]

Approvals (Signature/Date):

Technical Manager	Date 10/18/07	Health & Safety Manager / Coordinator	Date 10-15-07
Quality Assurance Manager	Date	Laboratory Director	Date 10/19/07

This SOP was previously identified as SOP No. CVS01504.CT.

Facility Distribution No. _____ Distributed To: _____

1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements

2.0 SCOPE AND APPLICATION

2.1 The TCLP is designed to determine the mobility of both organic and inorganic analytes present in solid, liquid and multi-phasic wastes.

2.2 If an analysis of any one of the liquid fractions of the TCLP extract indicates that a regulated compound is present at or above the regulatory level, the waste is considered to be hazardous.

2.3 The document control number for this SOP is CVS01504.CT.

3.0 TERMS AND DEFINITIONS

3.1 Refer to the SOP for Laboratory Term and Definitions.

4.0 SUMMARY OF METHOD

4.1 For wastes containing less than 0.5 percent solids, the waste, after filtration through a 0.8 um glass fiber filter, is defined as the TCLP extract.

4.2 For wastes containing greater than 0.5 percent solids, the liquid phase, if any, is separated from the solid phase and reduced by cutting, crushing, or grinding (if necessary), weighed, and extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid used is a function of the alkalinity of the solid phase. A ZHE (Zero Headspace Extractor) is used when volatile compounds are of concern.

4.3 If compatible, the initial phase of the waste is added to the liquid extract and analyzed following filtration. If incompatible, the liquids are analyzed separately and combined to yield a weighted average concentration.

4.4 This SOP is based upon EPA SW846 method 1311.

5.0 INTERFERENCES

5.1 Potential interferences that may be encountered during analysis are discussed in each of the individual analytical methods.

6.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

6.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against organic solvents.

The rotary extraction device should be checked daily before use.

The use of a vacuum system during sample filtration presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced. Ensure that the vacuum exhaust hose is vented to a fume hood so vapors are not pumped into the working environment.

Pressure may build up in the extraction vessel. Vent into a hood if needed.

6.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetic Acid	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.

1 – Always add acid to water to prevent violent reactions.
2 – Exposure limit refers to the OSHA regulatory exposure limit.

7.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

7.1 Samples should be stored unpreserved at 4°C, providing that refrigeration does not result in irreversible physical changes to the waste. Sample bottles are not reused.

7.2 When samples are to be analyzed for volatiles, care will be taken to minimize the potential loss of any volatiles.

7.3 The TCLP leaching for organic fractions must be performed within 14 days of sampling. Samples to be analyzed for mercury should be leached within 28 days, all other metals within 180 days.

8.0 APPARATUS AND MATERIALS

8.1 Top-loading balance, 0.01 g sensitivity

8.2 Agitation apparatus, 30 $\pm$ 2 rpm rotary mixer

8.3 Extraction vessels, glass or PTFE bottles with Teflon-lined caps, 2L capacity

8.4 Orion 720 pH/ISE meter with combination pH electrode or equivalent

8.5 Drying oven, capable of maintaining 100 $\pm$ 20°C or equivalent

8.6 Desiccator

8.7 Glass fiber filters (0.8 um)

NOTE: Glass fiber filters require rinsing with 1.0 N HNO_3 followed by rinsing three times with reagent water.

8.8 Pressure filtration apparatus

8.9 Vacuum filtration apparatus

8.10 Buchner funnels, 11 cm

8.11 Side arm filter flasks, 1 & 2 L

8.12 ZHE (Zero Headspace Extractors), [Figure 1].

8.13 Compressed Nitrogen Gas, ultra-high purity grade

8.14 Hot/stir plates

8.15 Beakers, 500, 150 mL

8.16 Metal spatula

8.17 Graduated cylinders, 1L, 100mL

8.18 Gas pressure

8.19 44mL glass volatiles vials

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Laboratory prepared reagent (nanopure) water is used whenever reagent ASTM II Water (ASTM D11930) is required unless otherwise specified.

9.2 For volatiles, organic free reagent water stored in the volatiles lab is to be used.

9.3 Hydrochloric acid (HCl), 1.0 N: Carefully add one part concentrated hydrochloric (reagent grade) to eleven parts reagent water and mix well.

9.4 Sodium hydroxide (NaOH), 6.0 N: Slowly and carefully add 240g concentrated sodium hydroxide pellets (reagent grade) to 1 liter reagent water.

9.5 Glacial acetic acid (HOAc), reagent grade

9.6 TCLP Extraction Fluid Concentrate purchased from Environmental Express; part number

E1002.

9.7 In case purchased TCLP Extraction Fluid Concentrate is unavailable the following recipe will be followed:
Extraction Fluid #1: Add 5.7 mL of glacial acetic acid and 10.7 mL of 6.0 N sodium hydroxide to approximately 800 mL reagent water. Dilute to 1L with reagent water. The pH of this fluid should be 4.93 ± 0.05. Add additional sodium hydroxide or acetic acid as necessary to adjust pH. For volatile extraction, a larger volume of 18L is prepared using 102.6mL of glacial acetic acid and 192.6mL 6.0N sodium hydroxide to 18L of reagent water. The pH is adjusted in the same manner as above to 4.93 ± 0.05.

Extraction Fluid #2: Dilute 5.7 mL of glacial acetic acid to 1 L with reagent water. The pH of this fluid should be 2.88 ± 0.05. Add additional sodium hydroxide or acetic acid as necessary to adjust pH.

10.0 CALIBRATION

10.1 Top-loading balance is calibrated daily to ± 0.01 g.

10.2 pH meter is calibrated prior to use with pH buffers 4, 7 and 10.

11.0 QUALITY CONTROL

11.1 All chemicals should conform to minimum specifications set by the Reagent Chemicals Committee of the American Chemical Society. All chemical inventories are used on a first-in first-out basis.

11.2 Employ a minimum of one blank per sample batch of 20 or fewer samples to determine if any contamination or any memory effects are occurring. Prepare a blank for each extraction fluid used. Prep blanks must be identified as described in the Classical Chemistry SOP for Labeling and Coding of Standards.

11.3 Matrix spikes are required for each waste stream. The analytical groups will spike leachates prior to any preservation, after the leaching process.

11.4 Each agitation apparatus (spinner) is checked monthly to verify that the device rotates at 30 ± 2 rpms (the number of revolutions is counted for one full minute). The verifications are

dated and recorded in the log book.

11.5 Record all NBS "S" weights where applicable.

11.6 ZHE's are checked for leaks both before and after TCLP rotary agitation.

12.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

12.1 Preliminary Evaluation: TCLP preliminary evaluations are performed on a minimum 100 g aliquot of waste, this aliquot shall not actually undergo TCLP extraction.

12.1.1 Percent Solids Determination

If the waste will obviously yield no free liquid when subjected to vacuum or pressure filtration through a 0.8 um filter, proceed to step 12.1.2.

If the waste is liquid or multi-phasic, separate the phases. Weigh out a representative subsample of the waste (approximately 100 g). Allow slurries to stand, to permit the solid phase to settle, if applicable.

Weigh out a 0.8 um glass fiber filter. Transfer the waste to the filter holder (prepared with the tared filter) and apply vacuum until all of the liquid has passed through the filter. Stop filtering when air passes through the filter. If this point is not reached, stop filtration when the liquid stops flowing.

Note: Oils which do not pass through the filter are treated as solids. Correct the initial sample mass for any material which adhered to the walls of the Millipore setup.

Remove the solid phase and filter media, while not allowing them to dry, weigh to ± 0.01 g. The wet weight of the residue is determined by the difference between the weight of the tared filter and the weight of the solid phase and filter.

Dry the filter and residue at 100°C ± 20°, and calculate the percent solids. The waste sample will be handled differently from this point depending on whether or not the waste contains more or less than 0.5 percent solids.

Note: This part of the procedure is used only to determine whether the solid must be extracted. Do not extract dried sample!

If the solid constitutes less than 0.5 percent of the waste, proceed to 12.3 for non-volatile parameters and 12.4.1 for volatiles.

12.1.2 Extraction Fluid Determination

If the solid content of the waste is greater than or equal to 0.5 percent and if non-volatile components are being extracted, the appropriate extraction fluid will have to be determined.

Note: TCLP extraction for volatile constituents uses only extraction fluid #1. If only volatiles are being extracted, proceed to 12.1.3.

Weigh out a small subsample of the solid phase of the waste, reduce (if necessary) to a particle size to 1 mm or less in diameter. Transfer 5.0 g of the solid to a 500 mL beaker. Add 96.5 mL of reagent water to the beaker, cover with a watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is <5.0, use Extraction Fluid #1, and proceed to step 12.1.3.

If the pH from step is >5.0, add 3.5 mL of 1.0 N HCl, slurry briefly, cover with a watchglass, heat to 50°C, and hold for 10 minutes.

Allow the solution to cool, and record the pH. If the pH is <5.0 use E.F. #1, or if the pH is >5.0 use E.F. #2.

12.1.3 Particle Size Determination

The solid phase of the waste must be crushed, cut, or ground in order that the solid may pass through a 9.5 mm or 0.375 in. standard sieve. If solids are being prepared for volatile fraction extraction, care must be taken to minimize exposure to the air, and prevent loss of volatiles. The generation of equipment blanks is performed to monitor for potential contamination from the particle size reduction equipment when particle size reduction is performed.

12.2 Non-Volatile Fraction Extraction (no free liquid)

12.2.1 If the waste will obviously yield no liquid when subjected to pressure filtration, weigh out a subsample of the waste which is compliant with step 12.1.3. and place the waste into an extraction vessel. A 100 gram sample should be used for the TCLP leaching.

12.2.2 Slowly add twenty times the mass of the waste of the appropriate extraction fluid (12.1.2) to the extraction vessel. The pH of the extraction fluid is measured and recorded prior to each use. Close the extractor bottle tightly, secure in the rotary agitation device, and rotate at 30 rpm ± 2 rpm for 18 hours ± 2 hours. Ambient room temperature shall be maintained during the extraction period (23°C ± 2°).

Note: Record the initial room temperature and final room temperature readings on prep. batch.

12.2.3 At the end of the extraction period, the leachate is filtered through a 0.8 um glass fiber filter. Leachates are placed in amber glass bottles for semi-volatiles, herbicides and pesticides; polyethylene bottles are used for metals.

12.3 Non-Volatile Fraction Extraction (with free liquid)

12.3.1 If the sample is liquid or multi-phasic, perform the liquid/solid separation. The liquid filtrate will constitute part or all of the extract.

12.3.2 Pre-weigh the container that will receive the filtrate. Assemble the filter holder and filter. Acid-washed filters may be used for all non-volatile extractions.

12.3.3 Weigh out a subsample of the waste and record the weight. If the waste contains <0.5 percent dry solids, the liquid portion of the waste, after filtration, is defined as the TCLP extract. Enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required of the TCLP extract. For wastes containing >0.5 percent dry solids, enough solids must be generated by filtration to support the analyses to be performed on the TCLP extract.

12.3.4 Quantitatively transfer the waste aliquot to the filter holder, and filter. If waste material (>1 percent) of the waste has adhered to the filter apparatus top, determine the weight of the residue and subtract it from the sample weight determined in Section 12.3.3 from the initial mass of the waste taken for extraction.

12.3.5 Quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase. Prepare the solid portion of the waste for extraction if required (see Section 12.1.3).

12.3.6 Determine the amount of extraction fluid to add to the extraction vessel (Section 12.3). Slowly add this amount of the appropriate extraction fluid. Close the extractor bottle tightly, secure in the rotary agitation device, and rotate at 30 rpm ± 2 rpm for 18 hours ± 2 hours. Ambient room temperature shall be maintained during the extraction period (23°C ± 2°).

Note: Record the initial room temperature and final room temperature readings on prep. batch.

12.3.7 Following the extraction period, the solution is filtered through 0.8 um glass fiber filters and placed unpreserved in bottles for the analytical groups.

Note: If any free liquid was collected prior to leaching, it should be combined with the leachate if compatible. If the two liquids are not compatible, they will be analyzed separately. The analytical results will be combined based on waste weight ratios following analysis.

12.3.8 Leachates are placed in amber glass bottles for semi-volatiles, herbicides and pesticides; polyethylene bottles are used for metals.

12.4 Volatile Extraction

12.4.1 Extracting Liquid Samples (<0.5% Solids)
Fill prepared, clean ZHE unit with liquid sample. Attach the nitrogen line, open the top valve, pressurize the unit expelling air from the unit. Close the top valve when all of the air has been expelled. Under pressure fill a minimum of three 44 mL volatile vials and label.

12.4.2 Extracting a Sludge Sample

Calculate the mass required to yield a 25g dry-weight sludge (see 13.4). Place the weighed waste aliquot into the ZHE unit and secure the lid. Attach the glass syringe to the ZHE unit using the Millipore interlock.

Attach the nitrogen line to the ZHE unit. Pressurize the unit to force the filterable liquid into the glass syringe.

Store the filtered liquid in 40 mL septa volatile vials at 4°C. If less than 40 mL of free liquid is available store the syringe containing the liquid at 4°C.

Attach nitrogen line to the unit containing the remaining non-filterable sludge; pressurize the unit to 1-10psi.

Add extraction fluid to the ZHE vessel using a pressurized reservoir. When adding fluid: open the valve located on the base of the ZHE unit, to release the pressure and allow the ZHE to fill with fluid. Once fluid stops flowing into the ZHE: close the bottom valve and the line from the reservoir. Invert the unit three times to bring unwanted air bubbles to the top; release the top valve to expel air and eliminate the bubbles. Re-attach the reservoir line and fill the remaining space left by expelling the air in the line. Pressurize the unit again to 1-10psi.

Place the ZHE in the rotary agitation device and spin for 18 hours ± 2 hours. At the end of the extraction period draw off the leachate through an in-line glass fiber filter via a glass syringe. Place the leachate into volatile vials and refrigerate at 4°C until the time of analysis.

Note: Record the initial room temperature and final room temperature readings on prep. batch.

12.4.2 Extracting a Solid Sample

Use the ZHE device to obtain a TCLP extract for analysis of volatile compounds only. The ZHE device has approximately a 500mL internal capacity and can thus accommodate a maximum of 25 grams of solid and extraction fluid equal to 20 times the weight of the solid (~500mL).

Charge the ZHE will sample only once and do not open the device until he final extract has been collected.

Do no allow the solid, the extraction fluid, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary.

Place the ZHE piston within the body of the ZHE (it may be helpful to first moisten the piston O-rings slightly with extraction fluid and use a rubber mallet to bang into place). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to travel once the ZHE is charged with sample. Place an O-ring into the groove on the top of the bottom flange and then place the ZHE body on top of it. Place an

O-ring into the grove on the bottom of the lid (top flange). Place a glass fiber filter between two support screens, place them over the O-ring in the top flange and secure with another O-ring. Set the lid aside.

Fill a ZHE unit with Extraction Fluid #1. Weigh out 25g ± 1g of sample and quickly transfer into the ZHE unit under the surface of the fluid. Secure the top flange immediately, tighten all the fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Attach a gas line to the gas inlet/outlet valve and begin applying pressure. Adjust pressure to 26psi.

Place the ZHE in the rotary agitation device and spin for 18 hours ± 2 hours. At the end of the extraction period, check the pressure behind the ZHE piston by observing the pressure gauge. If the pressure has not been maintained, the device is leaking. Check the ZHE for leaking and perform the extraction again with a new sample of waste. If the pressure within the device has been maintained, draw off the leachate through the liquid inlet/outlet valve on the top flange lid into at least two 44mL glass volatiles vials. Minimize air bubbles and air exposure by drawing off leachate on an angle to the inside of the vial. Fill the vials completely with no headspace and refrigerate at 4°C until the time of analysis.

Note: Record the initial room temperature and final room temperature readings on prep. batch.

13.0 CALCULATIONS

13.1 Percent Solids Calculation:

$$\text{Percent Solids} = \frac{\text{Weight of Solid Waste (g)} \times 100}{\text{Wet Sample (g)}}$$

13.2 Percent Dry Solids Calculation:

$$\%\ \text{Dry Solids} = \frac{(D-T) \times 100}{I}$$

where:

D = Weight of Dry Waste and Filter (g)

T = Tare weight of filter (g)
I = Initial weight of waste (g)

13.3 Amount of Extraction Fluid Calculation:

$$\text{Weight of Extraction Fluid} = \frac{20 \times \%\ \text{solids} \times \text{weight of waste}}{100}$$

13.4 ZHE Charge Calculation:

Weight of Waste to charge ZHE (g) = 2500/(% solids)

14.0 ACCEPTANCE OF DATA

14.1 Method blanks must not contain target analytes exceeding the practical quantitation limit (PQL) for that specified analyte. If a method blank fails surrogate recoveries or has a target analyte exceeding its PQL, then the QA manager is notified and a decision is made on how to correct the situation.

15.0 REPORTING OF RESULTS

15.1 All information is recorded in the TCLP preparation logbook.

16.0 POLLUTION PREVENTION

16.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

16.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

16.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

16.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

16.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

16.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

17.0 WASTE MANAGEMENT

17.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

17.2 **Waste Streams Produced by the Method**

The following waste streams are produced when this method is carried out.

- Acidic waste from sample extract. Employees are to collect and dispose of this material in satellite waste accumulation drums.
- Solid waste from sample extract. Employees are to collect and dispose of this material in satellite waste accumulation drums.

18.0 SUPPLEMENTAL DOCUMENTS

18.1 Classical Chemistry SOP for Labeling and Coding of Standards.

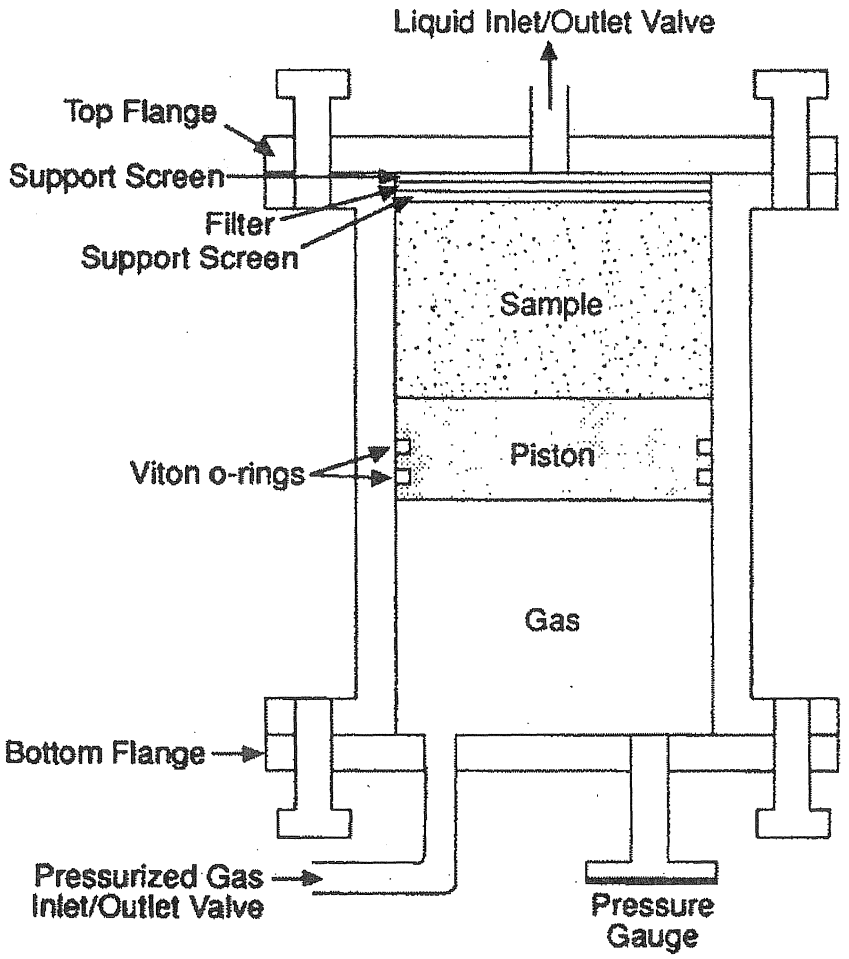
18.2 SOP for Laboratory Term and Definitions.

19.0 REFERENCES

19.1 Test Methods for the Evaluation of Solid Waste, SW846, 3rd Edition, Method 1311..

20.0 SUBSTANTIVE REVISIONS

20.1 Original document.



o Headspace Extractor (ZHE)

1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 The objective of this document is to outline the techniques for determining the presence and concentration of total hydrocarbons in multi-media, multi-concentration samples. The target compounds for this method are listed in Table 1.0. The method used in this procedure is a solvent extraction and gas chromatography/ flame ionization detector (FID) analysis.

2.2 The target compounds are those hydrocarbons found within the C9–C36 range.

2.3 The document control number for this SOP is CT-GCS-27, rev 4.

3.0 TERMS AND DEFINITIONS

3.1 There are many definitions used with in the laboratory, which may be generic to all laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used with in the laboratory, reference the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 This method employs the technique of solvent extraction using methylene chloride, coupled with a gas chromatography/FID analysis. An aliquot of sample extract is injected onto a gas chromatograph. A fused silica capillary column is then temperature programmed to separate the components prior to detection by FID.

4.2 This method is based on the document for the "Analysis of Extractable Total Petroleum Hydrocarbons (ETPH) Using Methylene Chloride Gas Chromatograph/Flame Ionization Detection.

4.3 Samples are prepared for analysis in accordance with methods SW846 3510C for aqueous samples and 3550B for solids.

4.4 Deviations

4.4.1 Reference "SOP for Prep of Aqueous Samples for DRO Analysis – 8015B" for procedure on extraction of Aqueous samples.

4.4.2 Reference " SOP for Prep of Soil Samples for DRO Analysis – 8015B" for procedure on extraction of Soil Samples.

5.0 INTERFERENCES

5.1 Interferences include all compounds, which are methylene chloride extractable and FID responsive. All materials used are demonstrated to be free from interferences by running laboratory method blanks.

5.2 Samples that contain high molecular weight compounds have a tendency to leave residues throughout the GC system, which may interfere with subsequent injection. Any samples which have chromatography indicating the possibility of carryover, will have solvent blanks analyzed after the sample injection, until the system has returned to normal.

6.0 HEALTH AND SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

6.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in both the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

6.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure

Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

7.1 Sample Containers

- Water samples are collected in 2x1 liter amber glass containers with Teflon-coated liner.
- Soil samples are collected in 250 or 500 mL glass containers with Teflon coated caps.
- Sample bottles are never reused.

7.2 Sample Collection

- Samples are secured against breakage in the shipping containers and kept at 4°C for transport to the laboratory. Samples should arrive at the laboratory the next day following collection.

7.3 Sample Preservation

- Samples are preserved by cooling to 4°C.

7.4 Holding Times

- Water samples must be extracted within 7 days from collection.
- Soil samples must be extracted within 14 days from collection.
- All extracts must be analyzed within 40 days from date of extraction.

8.0 APPARATUS AND MATERIALS

8.1 GC/FID/DS System

8.1.1 HP Model 5890 GC with FID Detector

8.1.2 Split/Splitless Injection Port

8.1.3 GC Column - DB-1, .53 mm ID Column or the equivalent

8.1.4 Perkin Elmer TurboChrom Data Aquistion System

8.1.5 Target software

8.2 Syringes - Various size 10 uL to 1 mL Hamilton or equivalent

9.0 REAGENT AND STANDARD PREPARATION

Solvent: Methylene Chloride, should be pesticide grade or equivalent

9.2 Alkane standard which contains a homologous series of n-alkanes for establishing retention times and the calibration curve.(C9 thru C36). The carbons included in this standard are C9, C10, C12, C14, C16, C18, C20, C22, C24, C26, C28, C30, C32, C34, and C36.

Table 1.0

Compound	Final Conc. (ug/ml) Individual Components	Final Conc. (ug/ml) Sum of Individual Alkanes
Alkane Mix (C9-C36)	6	90
	20	300
	40	600
	100	1500

200	3000
400	6000

9.3 All stock and working standards are stored in screw top bottles at room temperature and replaced after six months or earlier if necessary.

10.0 CALIBRATION

10.1 A five point calibration curve is prepared as described in Section 9. Calculate the average and relative standard deviation of response factors over the five concentrations. If the percent relative standard deviation (%RSD) of the response factor is < 30% over the working range, linearity through the origin can be assumed, and the average response factor (for the sum of the individual alkanes) can be used in calibration. If the %RSD cannot be met using the average calibration factor, an alternate curve type can be used. The criteria are 0.995 or higher for the coefficient.

10.2 Continuing calibration check standards, of a mid-level alkane standard, are analyzed each 12 hour shift, at a minimum. The percent difference for the average RF of the alkane standard must $\pm$ 30 percent difference from the average RF of the curve. The retention times of the individual alkanes and surrogate must fall within defined retention time windows.

10.3 If the continuing calibration criteria of $\pm$ 30% is not met, then a new calibration curve shall be analyzed.

10.4 Performance verification check standards, a mid-level alkane standard, should be analyzed at the beginning of a GC analysis batch and whenever any changes are made to the system or operational parameters. The percent difference for each individual alkane must $\pm$ 20 percent difference from the average RF of the performance verification standard. It is acceptable for up to one peak to be over 20%, but less than 35%. The retention times of the individual alkanes and surrogate must fall within defined retention time windows.

11.0 QUALITY CONTROL

11.1 Ortho-terphenyl shall be used as the surrogate(s) added to each sample, blank and QC.

11.2 An alkane standard consisting of C9 through C36 shall be used for the LCS, Matrix spike and Matrix spike duplicates. The LCS shall be extracted once per batch or every 20 samples and shall be from an alternate source than the calibration standards. The MS/MSD shall be extracted every 20 samples per matrix.

11.3 Calibration checks shall be analyzed each 12 hour shift with a $\pm$ 30 percent difference.

11.4 A Method blank is extracted each batch, per 20 samples.

11.5 An instrument blank spiked with o-terphenyl, surrogate, is analyzed after the continuing calibration standard, and prior to the analysis of any samples to determine that the instrument is free of contamination.

11.6 Analytical Documentation Procedures

11.6.1 Instrument Batches

An instrument batch is created for each analytical sequence to organize all the associated data. Batch designations are of the format:

XXnnn

where: XX = instrument identifier
nn = number of batch

(i.e. D3001)

Instrument batches are numbered sequentially so a unique batch identifier identifies each analytical sequence. The batch consists of a file folder with all the associated QC information for the analytical sequence. The raw data is then bound together with the file folder to complete the batch.

11.6.2 Filing System

All active batches are scanned to the server, any hardcopy printouts are discarded.

11.6.3 Data Archiving

All data files are archived on a daily basis using a 12.0gb data storage cartridge. The associated method files are also archived daily to provide an accurate historical record. Care shall be exercised when purging data off the hard drives to ensure that all data being purged has been archived.

11.6.4 Instrument Run Logs

It is STL's policy that all measurement data be recorded in logbooks or on preprinted log sheets in permanent ink. Run logs are created using a macro in the Target data system. This contains a sequential list of all files analyzed. It is signed and dated by the analyst. Corrections shall be made by drawing a single line through the error, and initialing and dating the correction. A secondary authorization of the logbook is required and shall be performed by the department's manager or designee.

Each instrument has its own set of run logs (see Figure 1.0) which are sequentially numbered and paginated. Run logs are filed in the laboratory once they have been filled, for future reference. Each analytical sequence shall be started on a new page of the log and continued on the next page, if necessary. The header information designating the standard codes used shall be completed for each sequence. All standards used are recorded in this field for future traceability. The data file, sample number, dilution factor, analyst's signature, and date are recorded.

11.7 Corrective Action Reports

A corrective action report (CAR) is initiated when a problem is encountered during analysis, data reduction or deliverables preparation, data validation, or when any deviations from this SOP occur (see Figure 2.0). The CAR is initiated by the analyst, or department manager first identifying the problem and is then submitted to the appropriate personnel including the QA Manager. Reference SOP for correction action reports.

The lower portion of the CAR is for the corrective actions taken, and is completed by the department or project manager when a corrective action decision has been made. The CAR is then redistributed to all the departments and individuals involved.

11.8 Chain of Custody Record

When samples are removed from storage for preparation or analysis they must be signed out utilizing the chain of custody record (COC). The samples shall then be signed back in on the COC upon their return to storage or designated "used" if the sample volume is consumed during the preparation or analysis.

11.9 Sample Tracking Record

Notification of sample arrival is done by the Sample Control department by issuing a preliminary notification sheet. Samples are tracked for extraction and analysis by using laboratory's LIMS system (Labnet).

11.10 MDLs are performed annually as per 40 CFR chapter 1.

12.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

12.1 Instrumental preparation:

Install appropriate column DB-1 manufactured by J&W or equivalent 30 m column.

Establish column flow rate at approximately 15 mL/min and column head pressure at 10 psi. Make sure the make up gas is fully open. Set hydrogen at 40 psi and air at 30 psi.

Light the FID detector. Wait about 10 minutes for the signal to stabilize. The flows can be adjusted to maximize sensitivity.

Set temperature parameters as follows:

Initial temperature: 40°C
Rate: 30°C/min.
Final time: 10 min.

Initial time: 0.5 min.
Final temperature: 310°C

Detector temperature: 300°C
Injector temperature: 290°C

12.2 Sample preparation:

Extracts are taken from the prep area and chain of custody is signed.

Then the extracts are loaded, under a hood, into 2 mL HP autosampler vials with 250ul inserts. Currently National Scientific HP5890 AS vials with screw caps are used but any equivalent is acceptable. Extracts are loaded using a new pasteur pipet for each individual sample to avoid any cross contamination. The autosampler vials are placed in the tray on the instrument.

The run sequences are programmed in the Perkin Elmer TurboChrom data system and the run is started.

12.3 Establishing the baseline

Column bleed is defined as the reproducible baseline shift that occurs during temperature programming of the GC column oven. To determine this area, a methylene chloride blank injection should be analyzed at the beginning of the day and after every 12 hours to determine the baseline response. This run is then used to perform baseline subtraction, using the TurboChrom Data System, in subsequent runs until the next methylene chloride blank is run.

12.3.1 The baseline for the sample is determined by beginning the integration at the desired start time (C9) and extending the baseline horizontally to the desired stop time (C36). A sum total area is used for calculations. See attached chromatograms for example integration and baseline criteria.

12.3.2 The integration is performed to incorporate the total area response between the C9 and C36 carbon range, established by running an alkane standard. This area should include all resolved and unresolved peaks. This total area must be adjusted to remove the area response contributed from column bleed (by using baseline subtraction) and from surrogates and peaks outside of the set retention time range (by subtracting manually).

12.4 Quantitation is performed by a summation of peak areas within a determined peak range based on the alkane standard.

13.0 CALCULATION

Water sample by average CF:

$$\frac{\text{(area sample) (final volume [uL])(dilution factor)}}{\text{(avg. Cf of Standard) (sample volume [mLs])(ul inj.)}} = \text{ug/L}$$

area/ng

Water sample by Linear regression:

b + (Area)/m1 = on column concentration (ng)

$$\frac{\text{(cn column concentration-ng) (final volume [uL])(dilution factor)}}{\text{(sample volume [mLs])(ul inj.)}} = \text{ug/L}$$

Water sample by Quadratic:

b + m1(Area) + (m2)*(Area)^2 = on column concentration (ng)

$$\frac{\text{(cn column concentration-ng) (final volume [uL])(dilution factor)}}{\text{(sample volume [mLs])(ul inj.)}} = \text{ug/L}$$

Soil sample by average CF:

$$\frac{\text{(area sample) (final volume [uL])(dilution factor)}}{\text{(average Cf of Standard) (wt [g])(dec. \% solids)(ul inj.)}} = \text{ug/Kg}$$

area/ng

Oil sample by average CF:

$$\frac{\text{(area sample) (final volume [uL])(dilution factor)}}{\text{(average Cf of Standard) (wt [g]) (ul inj.)}} = \text{ug/Kg}$$

area/ng

All areas are a summation of peaks for a determined time period which is consistent between the sample and standard.

14.0 ACCEPTANCE OF DATA

14.1 Method blanks should be free of any target compounds greater than the PQL.

14.2 The recovery of the LCS shall be within laboratory established control limits as listed in Table 2.0. If the recovery is out of criteria, reanalysis of the LCS must be performed. If the LCS is still out of criteria reextraction of the batch may be necessary.

14.3 The recovery limits for MS/MSDs extracted every 20 samples are listed in table 3.0. Reextraction is not required if recoveries are outside the QC limits unless the LCS failed control limits.

14.4 Daily calibration check standard verification of the calibration curve is required every 12 hours with the criteria of +/- 30 % difference.

15.0 REPORTING OF RESULTS

15.1 All results are reported to two significant figures. Water samples are reported in ug/L, soil samples are reported in ug/Kg dry weight and waste samples are reported in ug/Kg.

Check reporting deliverables required from the traveller. All job packages require a case narrative and quality control approval report. The case narrative should outline in detail any problems with client samples during analysis. The following indicates the different levels of reporting.

Level I

- Case Narrative
- Sample Results

Level II

- Case Narrative
- Sample Results
- Surrogate Recovery form
- LCS and MS/MSD recovery form
- Sample Scans

Level III/NJ

-All listed below, *Except* the standard scans and area reports

CLP/NYSDEC

- Case Narrative
- Form 1 (Organic Analysis Data Sheet)
- Surrogate Recovery form
- LCS and MS/MSD recovery form
- MSB recovery form as applicable
- Form 4C (Method Blank Summary)
- Initial Calibration Forms
- Analytical Sequence Form
- Continuing Calibration Forms
- Sample and Standard Scans and Area Reports
- Standard Concentration Summary

16.0 SUPPLEMENTAL DOCUMENTS

16.1 SOP for Extraction of Diesel Range Organics by Method 3510C.

16.2 SOP for Extraction of Diesel Range Organics by Method 3550B.

17.0 POLLUTION PREVENTION

17.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

17.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

17.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

17.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

17.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

17.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

18.0 WASTE MANAGEMENT

All waste shall be managed in accordance with all Federal, State, and Local requirements.

Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

18.1.1 Personnel who handle or generate waste must be trained within six months of employment in proper waste handling and requirements.

18.2 All autosampler vials containing methylene chloride as the main solvent should be disposed of in the 5 gallon bucket labeled for Hexane vial waste.

19.0 REFERENCES

19.1 Method 8015B, SW846, Test methods for Evaluating Solid Wastes, Third Edition including updates.

19.2 Method 3510C, SW846, Test methods for Evaluating Solid Wastes, Third Edition including updates.

19.3 Method 3550B, SW846, Test methods for Evaluating Solid Wastes, Third Edition including updates.

19.4 OQA-QAM-025-10/91, Quantitation of Semi-volatile Petroleum Products in Water, Soil, Sediment, and Sludge; 3/17/97, Rev. 3

19.5 State of Connecticut ETPH Method; Analysis of Extractable Total Petroleum Hydrocarbons (ETPH) Using Methylene Chloride Gas Chromatograph/Flame Ionization Detection. Prepared by Environmental Research Institute, University of Connecticut, March, 1999.

20.0 SUBSTANTIVE REVISIONS

20.1 Added to Safety section-2003.

20.2. Added to Waste management section-2003.

20.3 Revised Safety and Waste Management Sections to more Corporate standarized-1/2004.

20.4 Updated Tables with most recent Control limits-1/2004.

20.5 Changed Performance Verification Criteria from 30% to 20%.

20.6 Updated sections 11.6.2, Filing system and section 11.6.4, run logs and 11.7 CARs: 9/9/06

TABLE 2.0

LCS CONTROL LIMITS

Compound	Soil	Water
Alkane C9-C36	55 - 133%	52 - 128%

TABLE 3.0

MS/MSD CONTROL LIMITS

Compound	Soil	Water	RPD
Alkane C9-C36	55 - 133%	52 - 128%	20%

TABLE 4.0

SURROGATE CONTROL LIMITS

Compound	Soil	Water
o terphenyl	24-142%	64-125%

TestAmerica Anchorage
2000 W. International Airport Road
Suite A10
Anchorage, AK 99502
phone 907.563.9200 fax 907.563.9210

Chain of Custody Record

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Laboratories, Inc.

Client Contact	Project Manager:	Site Contact:	Date:	COC No:
Your Company Name here	Tel/Fax:	Lab Contact:	Carrier:	_____ of _____ COCs
Address	**Analysis Turnaround Time**			Job No.
City/State/Zip	Calendar (C) or Work Days (W) _____			
(xxx) xxx-xxxx Phone	TAT if different from Below _____			
(xxx) xxx-xxxx FAX	☐ 2 weeks			SDG No.
Project Name:	☐ 1 week			
Site:	☐ 2 days			
P O #	☐ 1 day			

Sample Identification	Sample Date	Sample Time	Sample Type	Matrix	# of Cont.	Filtered Sample																	Sample Specific Notes:

Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other _____

Possible Hazard Identification

☐ *Non-Hazard* ☐ *Flammable* ☐ *Skin Irritant* *Poison B* ☐ *Unknown* ☐

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)

☐ *Return To Client* ☐ *Disposal By Lab* ☐ *Archive For _____ Months*

Special Instructions/QC Requirements & Comments:

Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time: